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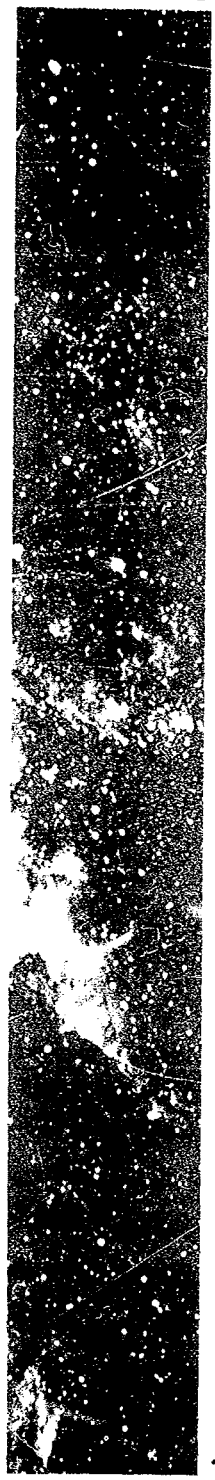

BEHAVIOR OF MATERIALS IN VACUUM

FINAL REPORT

GENERAL ELECTRIC

DIN 68SD4333

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IN VACUUM

FINAL REPORT

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ABSTRACT

Among problems which have been encountered in ground testing and flight of spacecraft have been those related to the outgassing of materials in vacuum. One of the major problems caused by outgassing is its condensation onto critical surfaces. The surfaces of a spacecraft most susceptible to damage by condensation of a foreign material are those used in optical systems. This report describes the techniques used to determine the effects of outgassed materials condensed on MgF_2 overcoated aluminum mirrors while the mirrors were irradiated with ultraviolet light in vacuum. Mirror temperatures as low as $-60^{o}C$ were used to determine the point at which condensation was incipient. The measurements performed on these mirrors included reflectance at Lyman α (1216 Å) and infrared analysis of the deposit. The materials investigated include: aluminized Mylar, RTV 60 and Sylgard 184 silicone rubbers, Raychem Specification 44 Spacewire, plastic gloves, and silicone grease. Some of these materials produced large amounts of condensate while others produced little or no condensate.

SECTION 1
INTRODUCTION

Some space vehicles have experienced a fogging of optical surfaces in ground based simulation and/or in space flight. This fogging (condensate) in ground testing is due to materials sublimed from the space vehicle, the chamber proper and associated fixturing, wiring etc., while in flight it is due to material sublimed from the space and launch vehicle both during launch and after injection into orbit or interplanetary flight. It has recently been noted that the outgassed products and debris which leave the vehicle while it is in orbit tend to "follow" the vehicle rather than go off immediately into deep space[1]. If material condensed onto critical surfaces of the vehicle it could be detrimental or even catastrophic to the mission. A change in the surface characteristics of critical surfaces resulting either from the condensed film itself and/or the interaction of the condensed film with the surface on which it condenses could cause mission failure. This phenomenon is further complicated by the potential interaction of the condensate and/or interacted surface with the radiation components of the space environment, not to mention manmade radiations such as high altitude nuclear blasts and nuclear power systems. Since most critical surfaces, such as optical elements and thermal control surfaces, will be on the exterior of the spacecraft, the most important natural space radiations will be near and far ultraviolet radiation, low energy protons, and low energy electrons. In ground testing there are additional critical surfaces on the test equipment; such as, solar simulator optics and colliminator and target surfaces used in testing.

The work performed in this study represents an initial investigation into this problem. It is believed that this is the first time the effect of outgassed products on the Lyman α reflectance of mirrors was investigated while under near ultraviolet radiation in thermal vacuum. The environmental exposure technique utilized, incorporates maximum sensitivity to condensation and known solid angles from the sample to the condenser necessary for using the results in quantative analysis for other systems. The techniques for quantitative analysis for very minute condensates have been used successfully in this program. This consisted primarily of frustrated multiple internal infrared reflectance of the condensed

films on the mirrors. The environmental exposure consisted of nominally 1000 equivalent ultraviolet sun hours (EUVSH)* of near ultraviolet radiation at an intensity of nominally 10 equivalent ultraviolet suns (EUVS)** while the mirror condensers were in a vacuum in the low 10^{-6} torr region at temperatures from $+60^{o}$C to -60^{o}C.

The materials investigated included: 1/4 mil NRC-2 one side aluminized Mylar (crinkled), General Electric RTV-60 and Dow Corning Sylgard 184 silicone rubbers, Raychem Specification 44 Spacewire, poly (vinyl chloride) gloves, and Dow Corning high vacuum silicone grease. Of the materials investigated the poly gloves and the silicone rubbers produced the most condensate and should be restricted or eliminated in their use on space chambers and spacecraft. In the process of developing a satisfactory facility and suitable analytical techniques for this investigation a number of obstacles were encountered. The solution to these obstacles resulted in some surprising discoveries. Among them were; an increase in the Lyman α reflectance of some MgF_2 overcoated aluminum mirrors when exposed to near ultraviolet radiation and a transmission window at Lyman α (1216 $\mathring{A}$) in methyl silicone.

A number of modifications which should be incorporated in future programs have come to light in the process of performing this work and other related programs involving simulation of the space environment for thermal control coatings. These modifications are presented in Section 7, Recommendations for Future Work.

*EUVS - Equivalent Ultraviolet Sun(s) for this program is defined as the total electromagnetic energy between 0.25 and 0.40 microns at zero air mass and 1 AU. This is given in the Johnson[2] curve.

**EUVSH is the hours of equivalent exposure to this radiation.

SECTION 2
TECHNICAL APPROACH

Numerous methods have been used to determine the effect of outgassed materials condensing onto optical surfaces. Some have attempted to simulate the hardware geometry, while others just used a direct line of sight between the optical surface and the material under study. The tests which simulated hardware conditions solved only the specific problem which was under study, and sometimes not even that if the proper assumptions and simulations were not used. The data obtained in this manner could not usually be generalized to other cases. When direct line of sight methods are used, one has little knowledge of the true view factor (because of fixtures, etc. from which outgassed particles can be reflected) and usually obtains insufficient condensate for analysis.

A suitable test method should include at least the following:

a. A simple geometry that will allow the data obtained to be used in the analysis of any case desired.

b. A geometry such that the maximum possible condensables outgassed from a particular sample can be collected.

The "distribution box" technique described below satisfies both these requirements.

2.1 DISTRIBUTION BOX TECHNIQUE

The test configuration for the distribution box technique consists of a setup as shown in Figures 2-1 through 2-3. The sample is placed onto the lid of a cylindrical box whose bottom has only one hole (centered directly over the two mirrored surfaces). The sample is then heated to +60^{o}C by the radiation from the box which is heated with infrared lamps. The temperature of the sample is regulated with a thermocouple imbedded in it such that the sample is always less than or equal to the minimum box temperature which is obtained from numerous thermocouples mounted on it. The outgassed material is directed onto the two mirrors through the hole cut into the bottom of the box. One condenser (mirror) is maintained at -60^{o}C throughout the test so that it will gather the maximum possible

condensate. The temperature of the other mirror is kept at +60°C until a condensate is seen forming on the -60°C mirror. At this time the temperature of the mirror which was at +60°C is slowly lowered until a deposit begins to form. This operation establishes the temperature above which a condensate will not form under the given conditions. Both mirrors are irradiated with the ultraviolet radiation during the entire test, this is begun before the specimen temperature is raised from room temperature and as soon as the mirrors are at temperature. The ultraviolet radiation is directed onto the mirrors, since some of the condensables may be polymerized by the photons in the ultraviolet region of the spectrum. There is sufficient reason to believe that this could happen since the condensables outgassed from a large number of spacecraft and space chamber materials will be monomers, low molecular weight polymers, plasticizers, and excess catalysts. Many of these materials might condense and resublime if they were not polymerized by ultraviolet radiation. It is known that many materials will polymerize (cross-link) under ultraviolet radiation.[3] Once a material has poiymerized, its outgassing (sublimation) rate decreases considerably due to the formation of higher molecular weight material with a different (lower) vapor pressure.

With this particular geometry, 64 percent of the particles which pass through the hole in the box will hit the mirror. The sticking factor can be obtained approximately by measuring the weight gain of the mirror and the weight loss of the sample.

To make the test as realistic as possible the sample thickness used was similar to that which is normally used in actual applications. The integrated energy in the spectrum between 2500 and 4000 Å was about ten times that in the Johnson curve between these same wavelength intevals; therefore, each actual test of 1000 equivalent ultraviolet sun hours required about 100 hours of irradiation.

The spatial intensity, Figure 2-4, and spectral distribution, Figure 2-5, of the ultraviolet source at the mirror site was established with an Eppley thermopile and a set of calibrated Eppley bandpass filters. Since the majority of materials used in a spacecraft and in the simulation chamber will not be exposed to ultraviolet radiation in actual use, the hole in the bottom of the distribution box was positioned so that virtually no radiation was reflected from the mirrors to the sample.

The principle by which the distribution box increases the condensate on the mirrors is by enabling large sample surfaces to have a large effective view factor to a small mirror surface. This is obtained by forcing the outgassed material from a large sample surface to exit the distribution box only through a relatively small hole which has a large view factor to a small mirror. Problems associated with outgassed material from the sample condensing on the inside walls of the distribution box are eliminated by maintaining the walls of the box at a temperature slightly greater than or equal to the sample temperature. All other sources of condensables were eliminated by using only metals, quartz, glass, ceramics, and Teflon in the test system. The mirror substrates used were fabricated from a ferrotype plate substrate (used in photographic processes for making glossy prints) with an evaporated aluminum mirror and an overcoating of magnesium fluoride. A metal plate was used as the mirror substrate to allow positive attachment of the mirror (with screws) to the temperature control platen, thereby eliminating uncertainties in the surface temperature of the mirror due to variations in contact resistances between different mirrors and the control platen.

2.2 REFLECTANCE AT 1216 Å

The reflectance of all the mirrors used in this study was measured at 1216 Å prior to their exposure to contaminants in vacuum. Three of these mirrors were kept as reflectance standards for use throughout the program. One of the standards was normally kept in the reflectance measurement apparatus, one in a suitable container in air, and the third in an evacuated sealed tube. While such precautions may have been unnecessary, they assured reliable reflectance standards throughout the program.

The reflectance of a sample mirror was measured relative to the standard just prior to its exposure to contaminants in vacuum. Upon completion of the exposure, its reflectance was again measured relative to the same standard.

Reflectance at 1216 Å was measured with a Jarrell-Ash Seya-Namioka vacuum monochromator, Figure 2-6. The reflectance attachment shown schematically in Figure 2-7 was used to measure reflectance at an incident angle of 45°. Measurement of reflected light intensity was made with an EMI 6094B photomultiplier, having a thin deposite of sodium salicylate on the window as a scintillator.

The light source used for the 1216 Å reflectance measurements was a microwave activated hydrogen discharge lamp. The lamp is filled with 2 to 4 torr of a mixture of 90 percent argon, 10 percent hydrogen. A lithium fluoride window is attached to the lamp with a silver to silver chloride seal. Such lamps have proven to be quite stable and to have a relatively high output at 1216 Å.

2.3 ANALYSIS OF MIRROR CONTAMINANTS

Mirrors which were exposed in the vacuum system were weighed before and after exposure. Those mirrors which showed appreciable change in reflectance at Lyman α were also subjected to infrared analysis by frustrated multiple internal reflectance (FMIR). In addition, FMIR measurements were performed both before and after environmental exposure, in order to elucidate the cause of Lyman α reflectance increase. Infrared analysis of many standards, against which various condensates could be compared, were also performed.

2.4 CHANGE IN SAMPLE WEIGHT

The samples were pre- and post-conditioned in a controlled environment of 75 ± 3°F and 43 ± 3% relative humidity. The difference between the pre- and post-conditioned weight was used to compute the percent weight loss of the material tested. This weight loss is referred to as the "bulk" weight loss since it is only the material per se, i.e., the absorbed and adsorbed moisture and gas loss was canceled out by the sample conditioning technique.

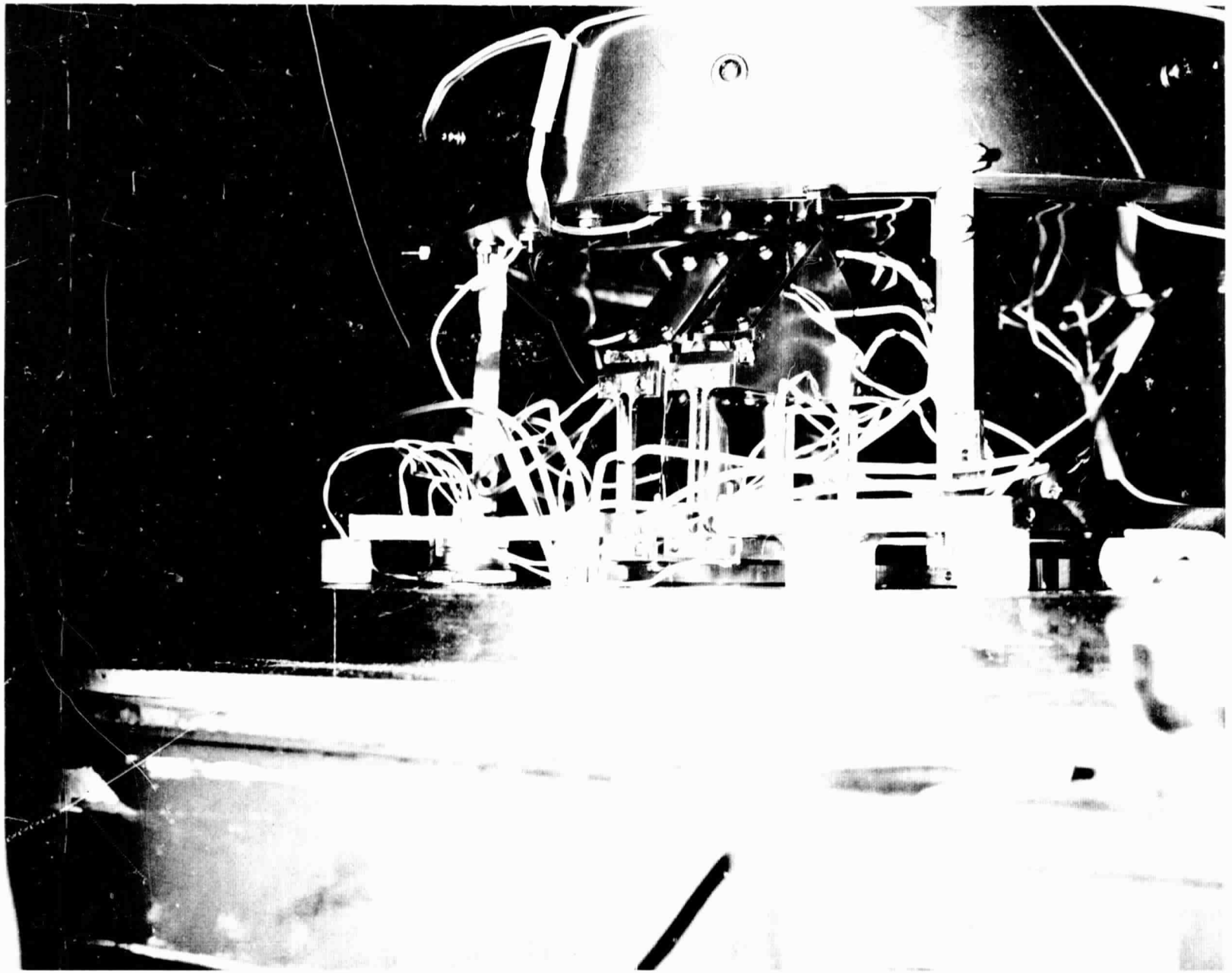


Figure 2-1. Mirror Platens and Bottom of Distribution Box

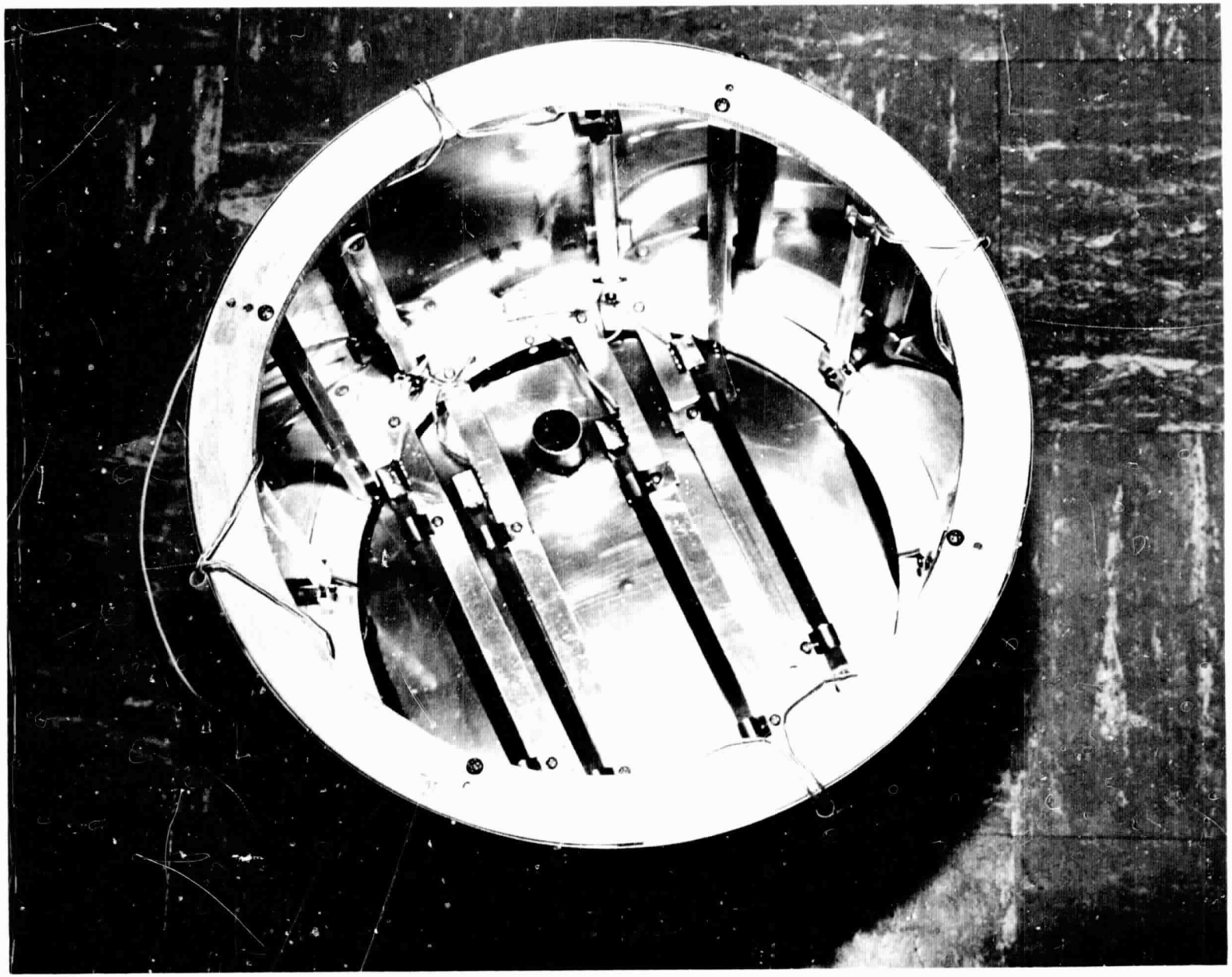


Figure 2-2. Inside View of Outer Shell of Distribution Box

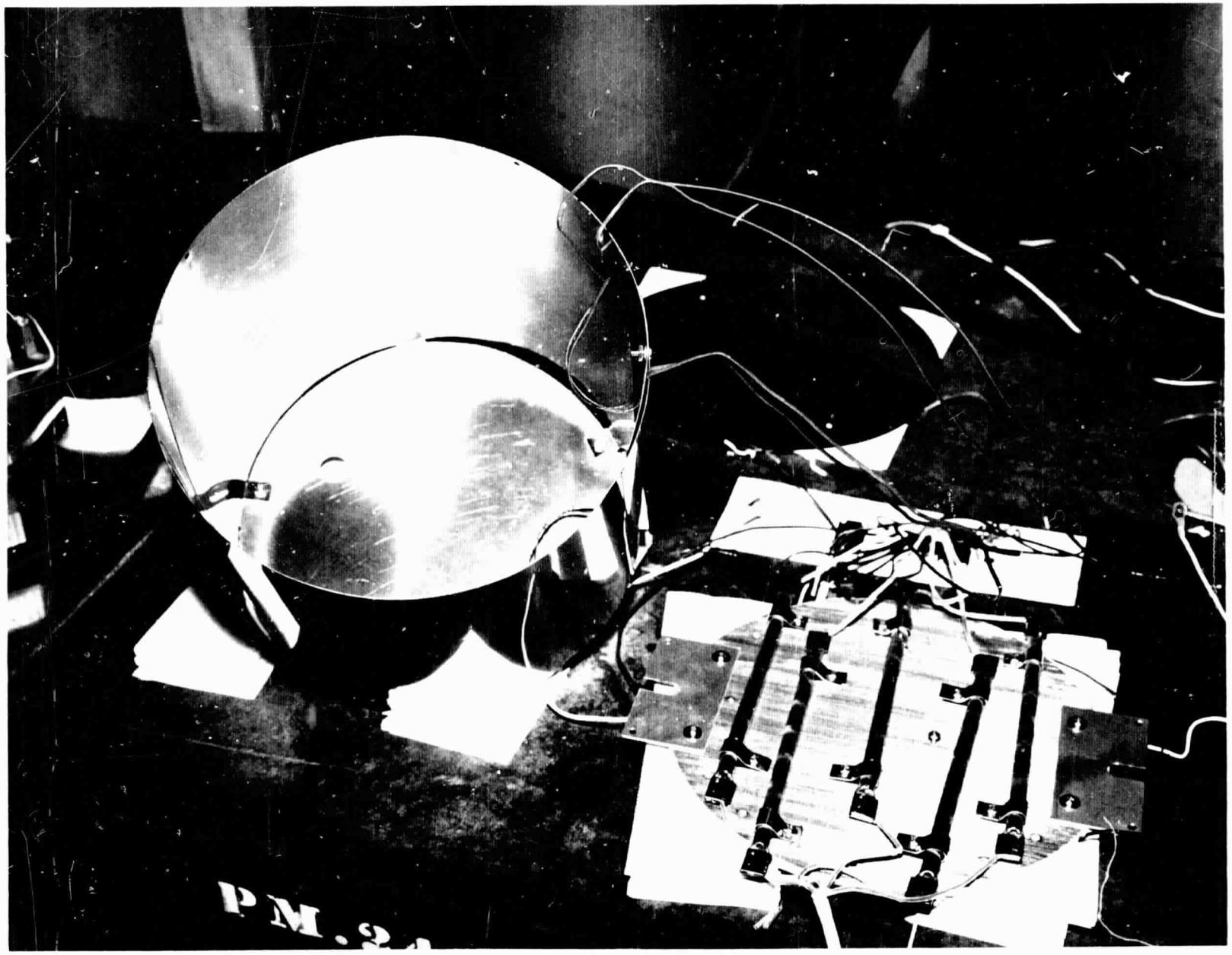
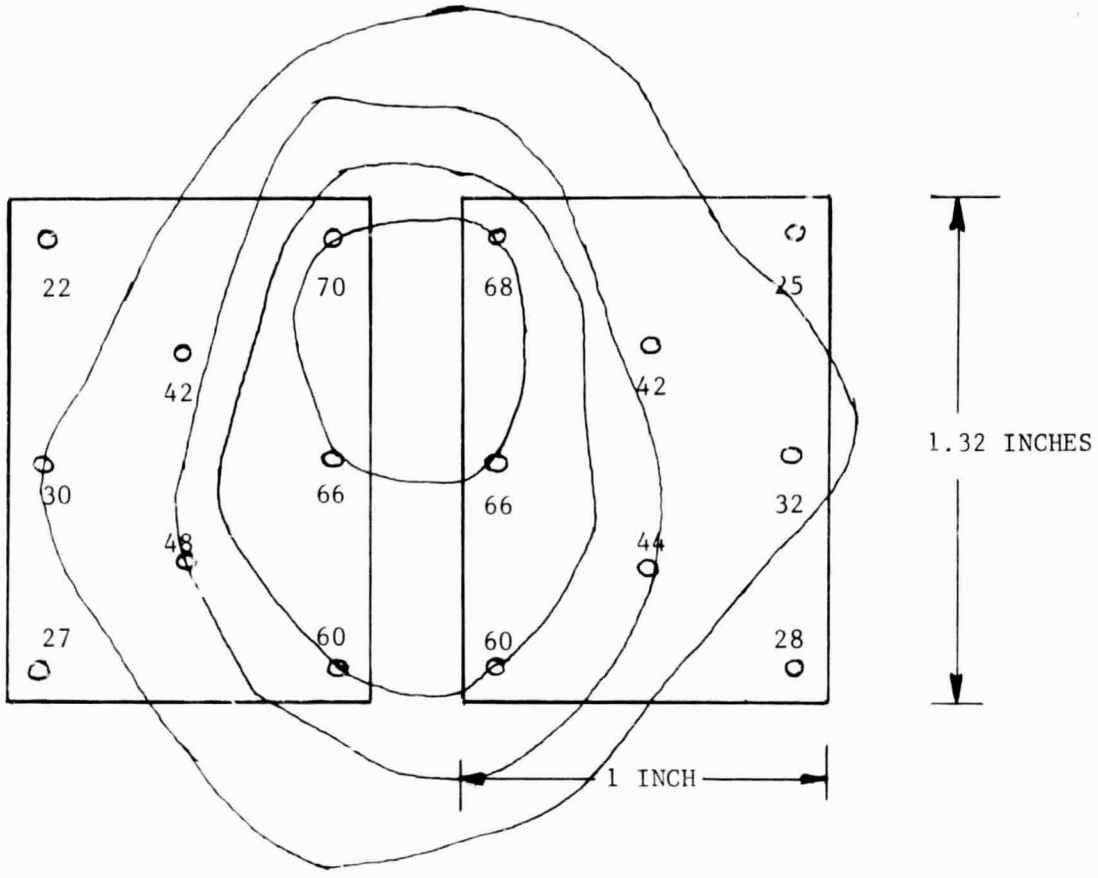
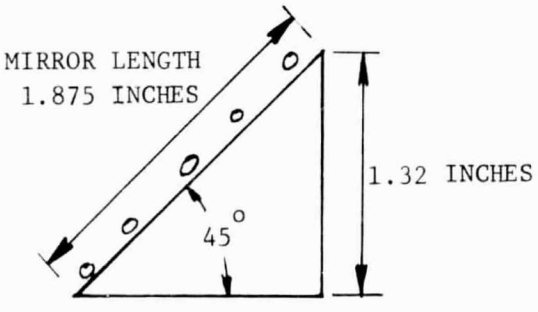


Figure 2-3. Inside Shell of Distribution Box and Lid





VALUES INDICATED ABOVE ARE THE THERMOPILE MILLIVOLT READINGS AT POSITIONS CORRESPONDING TO THE LOCATIONS ON THE INCLINED MIRROR SURFACE AS SHOWN

Figure 2-4. Spatial Intensity Plot of Radiation Intensity in Sample Plane

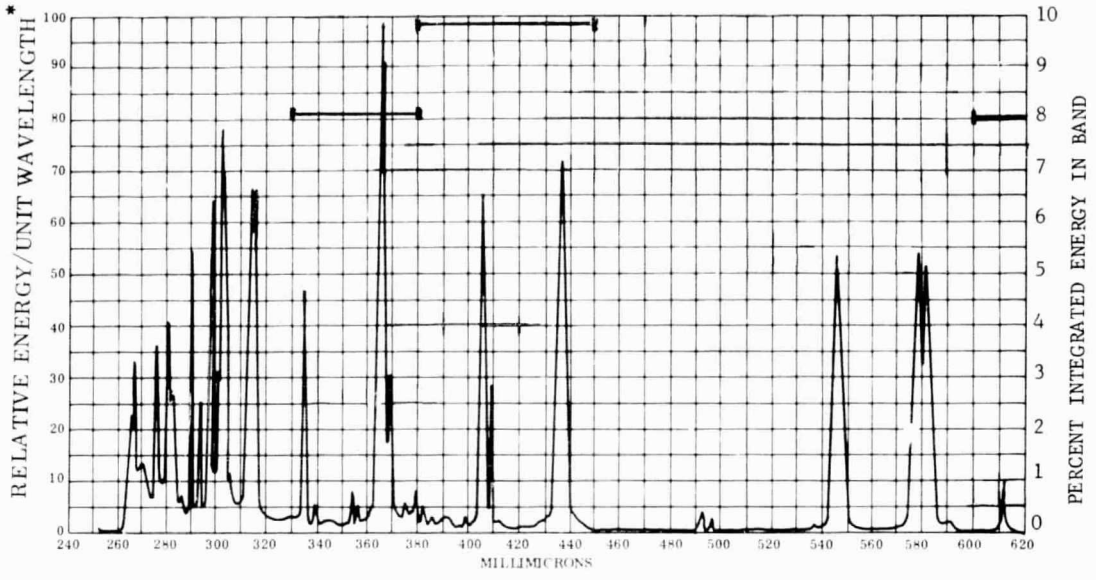
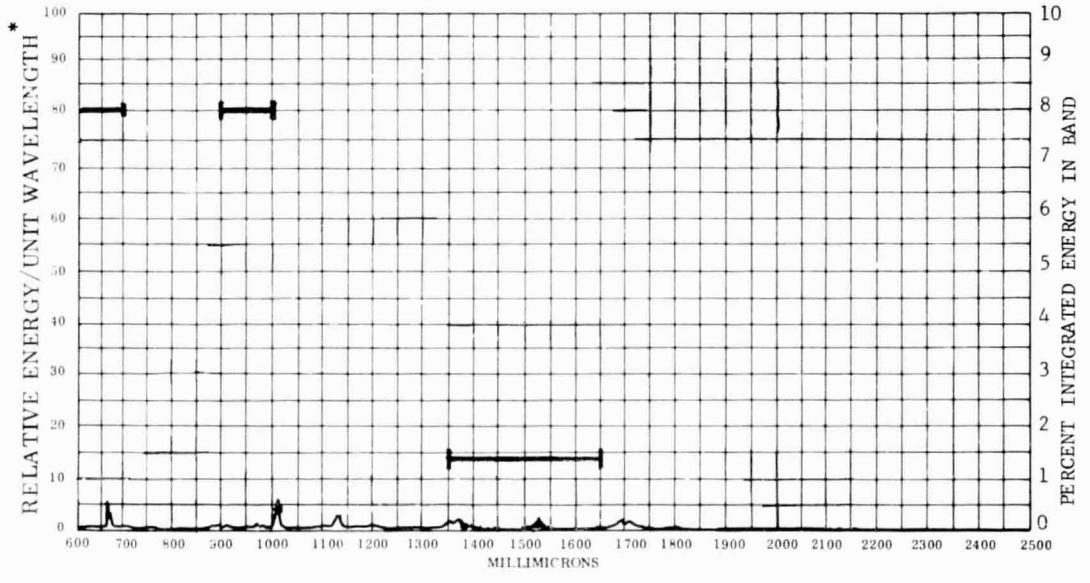


Figure 2-5a. Relative Spectral Irradiance



*"REPORT ON AN EXAMINATION OF SPECTRAL EMISSION CHARACTERISTICS OF XENON AND MERCURY XENON SHORT ARC LAMPS," EPPLEY LABORATORY INCORPORATION, JULY 1962.

⊢—⊣ PERCENT INTEGRATED ENERGY IN BAND MEASURED
—— (CONTINUOUS CURVE) RELATIVE ENERGY PER UNIT WAVELENGTH

Figure 2-5b. Relative Spectral Irradiance

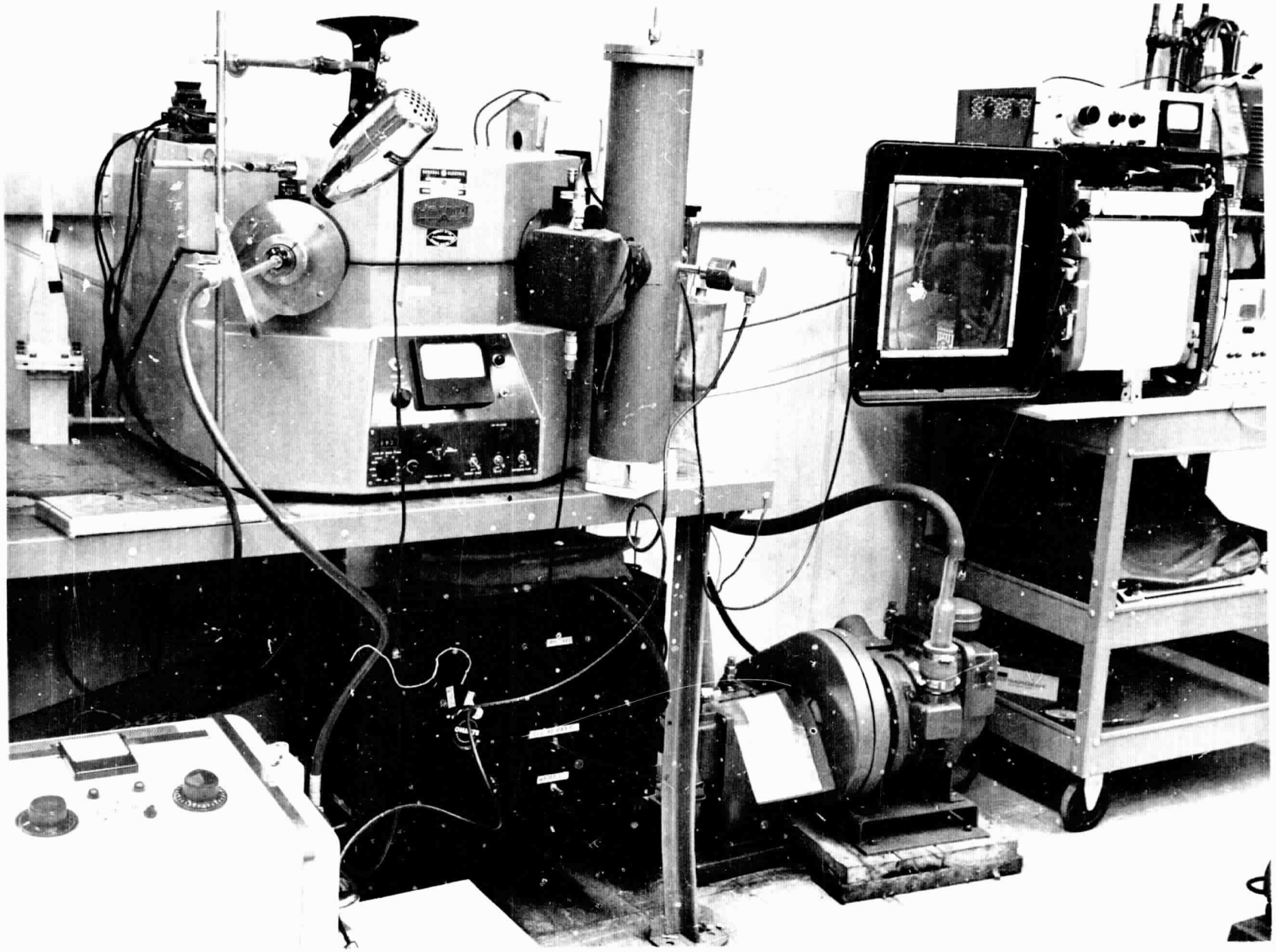


Figure 2-6. Lyman α Reflectance Facility

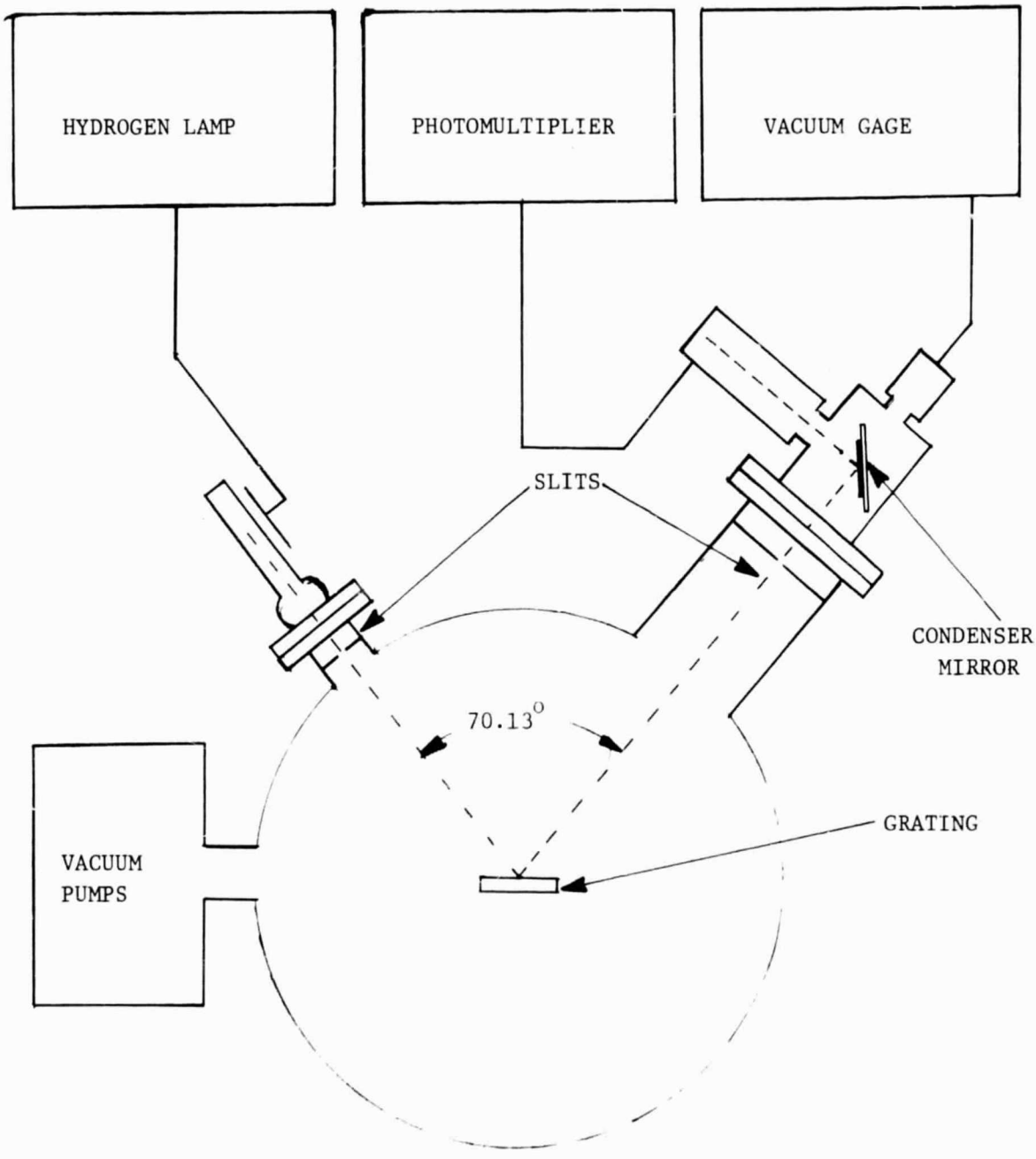


Figure 2-7. Schematic of Lyman α Reflectance Facility

SECTION 3

ENVIRONMENTAL EXPOSURE FACILITY

A photograph of the environmental exposure facility is shown in Figure 3-1. The vacuum system is fabricated of 304 SS and is 18 inches in diameter and 18 inches high. The facility is shown schematically in Figure 3-2. It is evacuated with a 750 liter/sec NRC HS-750 diffusion pump backed (and roughed) with a 3.3 cfm E. Ligbold's Bachfolger mechanical pump. The diffusion pump is separated from the base plate with a CVC anti-migration chevron optical baffle trap which is cooled with liquid nitrogen. A high vacuum gate valve is used above the chevron trap. The common part of the mechanical pump line is trapped with a water cooled optical baffle. The pressure is measured with an inverted Bryant-Alpert type ionization gauge which is mounted in the tubulation between base plate and the high vacuum gate valve. Thermocouple gauges are installed in the base plate and the backing line.

3.1 EVACUATION PROCEDURE

The diffusion pump is normally kept under high vacuum even when the vacuum bell is open. The system is operated by roughing the bell out and then opening the high vacuum valve after the trap has been cooled. This procedure has been successful in eliminating all trace of any backstreaming diffusion pump fluid onto the condenser mirrors. The platens are brought to the test temperature, $\pm 60^{o}C$, and the UV lamp is turned on as soon as the vacuum system is in the 10^{-5} torr range. The distribution box and the test sample inside it are then heated to the test temperature of $+ 60^{o}C$. The system is shut down by closing the high vacuum gate valve, turning off the UV lamp and distribution box heater power, and then bleeding in dry gaseous nitrogen to about 10^{-2} torr. After the distribution box has reached room temperature the condenser platens are returned to room temperature and the vacuum system is returned to atmospheric pressure with dry gaseous nitrogen.

3.2 DISTRIBUTION BOX

The distribution box shown in Figures 2-1 through 2-3 is constructed of 304 SS (except for an aluminum lid) and is heated with 500/T-3 General Electric infrared heating lamps.

These lamps are trimmed to power with powerstats and are controlled with API/thermocouple controllers. All wiring on the lamps and thermocouples is Teflon coated. The temperature at various locations on the distribution box (both inner and outer) is recorded. Measurements of the temperature profile of the distribution box showed that it is uniform to $\pm$ 2.5^{O}F under steady state conditions. The hole in the bottom of this box subtends a 50 degree solid angle to the condenser mirrors located beneath it.

3.3 CONDENSER PLATENS

The condenser platens are machined from copper as shown in Figure 3-3. They contain a passage for gaseous/liquid nitrogen cooling, in addition to tungsten heating elements. The temperature is controlled with copper-constantan thermocouples soldered into the platen surface (under the center of the mirror). One thermocouple is used for recording and the other for the API controller. The controller is an on/off/on type which calls for either power to the heating elements or liquid nitrogen to the cooling duct located in the platens, depending on the platen temperature in relation to the controller set-point. The mirrors are bolted to the platens with one screw in each corner. All wiring for power and thermocouples of these platens is Teflon insulated.

3.4 MIRROR MEASUREMENT SEQUENCE

The Lyman α reflectance of the mirrors is measured immediately before they are to be exposed to thermal vacuum and ultraviolet radiation. About half way through the program we also started measuring the FMIR infrared spectra of each mirror before environmental exposure but after the initial Lyman α reflectance. The purpose of this was to help elucidate the reason for the increases of the Lyman α reflectance of some mirrors due to environmental exposure. As soon as possible after environmental exposure (usually within one day) the Lyman α reflectance of the mirrors is remeasured. The FMIR infrared spectra of the mirrors are obtained after Lyman α reflectance.

3.5 ENVIRONMENTAL CONDITIONS

After a vacuum of 10^{-5} torr is obtained the mirrors are brought to their initial test temperatures of +60^{O}C and -60^{O}C. Once these are obtained the 2.5 Kw HgXe lamp is started and

set to obtain an average intensity of 10 EUVS on the mirrors. The distribution box is then heated so that the sample inside it is at +60°C with the inside of the box at +60°C or greater. The test duration is 1000 EUVSH, which requires nominally 4 days of environmental exposure (100 hours). As soon as a readily perceptable film is noted on the cold mirror (-60°C), the hot mirror temperature (originally +60°C) is lowered in 10°C increments until a film formation is visible on it. It is then maintained at that temperature for the duration of the test. Each temperature level below +60°C is maintained for at least four hours before being lowered again.

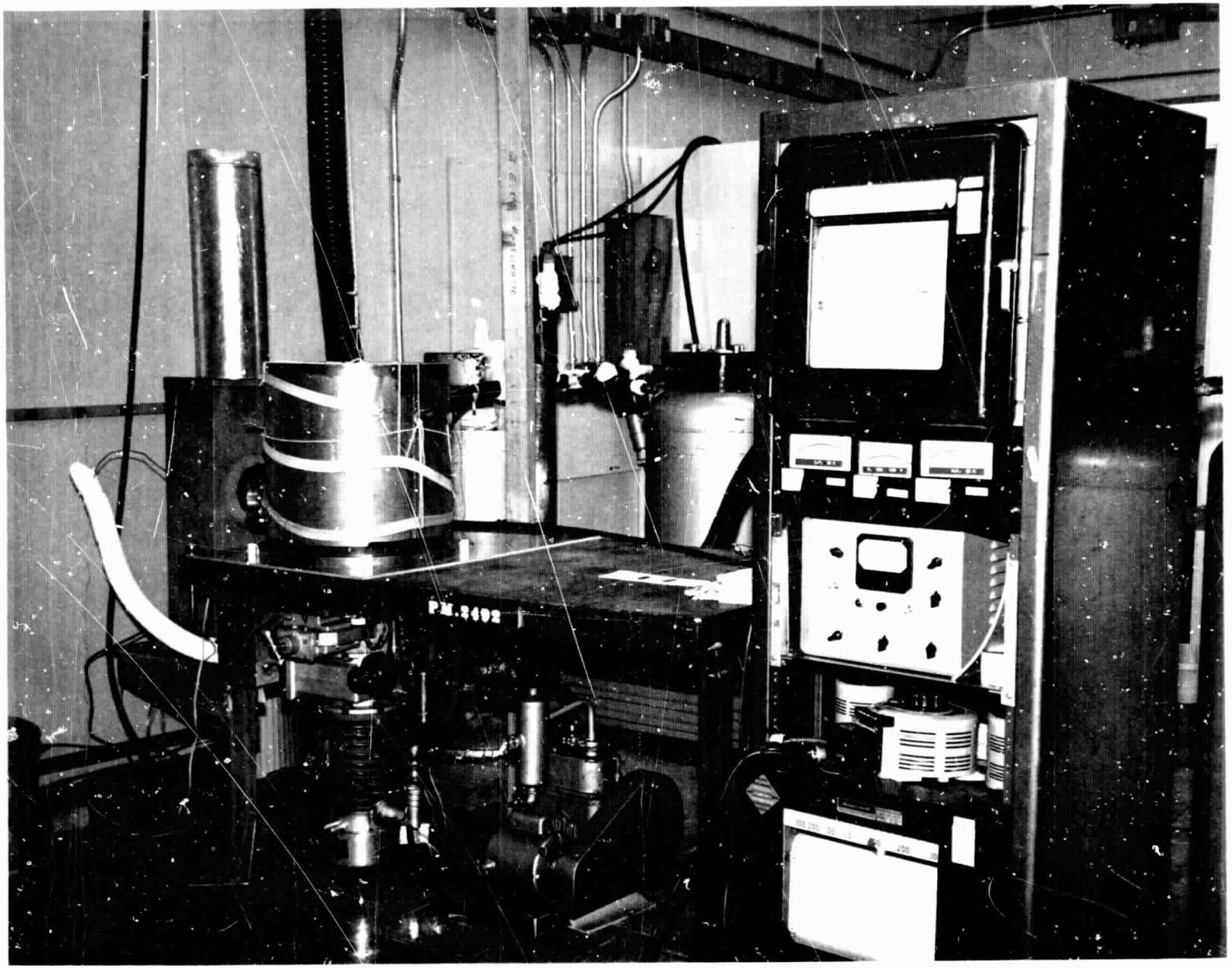


Figure 3-1. Thermal Vacuum/Irradiation Exposure Facility

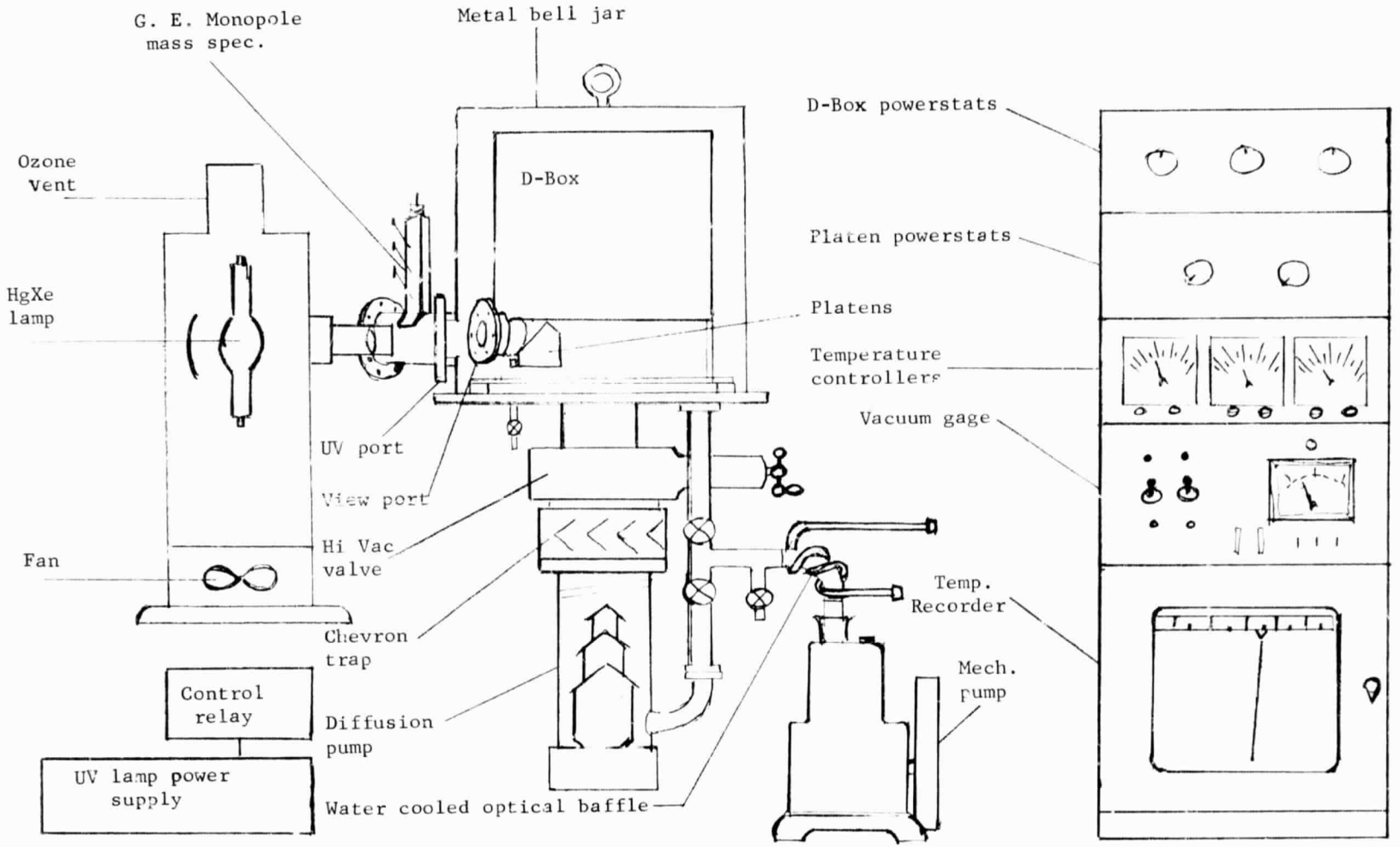


Figure 3-2. Schematic of Environmental Exposure Facility

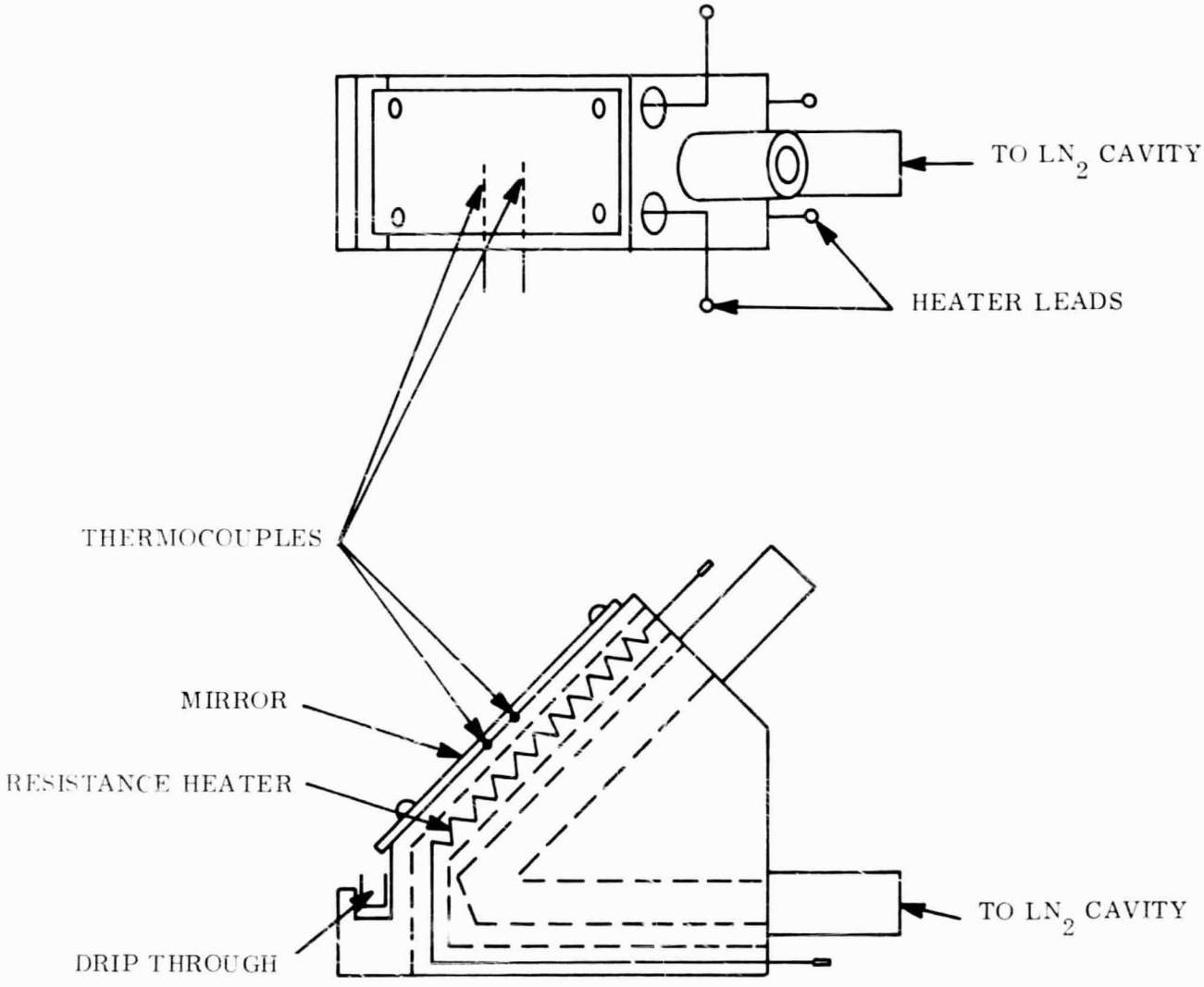


Figure 3-3. Mirror Platen Schematic

SECTION 4

EXPERIMENTAL PROGRAM

The facility was fabricated and the ultraviolet radiation source and distribution box were calibrated as described in Sections 2 and 3. The temperature gradient through the mirror was then measured by irradiating a mirror with a thermocouple epoxied to its front surface. The epoxy was coated with vapor deposited aluminum to obtain optical characteristics similar to that of the mirror. The thermal gradient at steady state conditions was $3^{\circ}C$ maximum with the mirror at $-60^{\circ}C$ (the front surface being warmer). After the entire system was baked out, a control test was conducted. A control test is identical to a test run as described in Section 3, except that no sample is placed in the distribution box.

The first control tests conducted resulted in very light condensates being formed on the cold ($-60^{\circ}C$) mirrors. Subsequent short "control tests" (less than four days and/or without ultraviolet radiation) combined alternately with numerous bake outs and solvent cleanings of the system resulted in the discovery that the condensate was being caused primarily from plasticizer residue being left on the chamber parts due to solvent cleaning with plastic gloves. Infrared spectra of solvent leachings from the gloves and of the mirror condensates proved to be identical, dioctyl phthalate. Subsequent infrared spectra of solvent leachings of numerous types of flexible "nonpermeable" gloves proved that all types tested leached some organic material. Among the gloves investigated were surgeon's gloves and polyethylene throw-aways. After this, the chamber and internal parts were cleaned with sterile gauze saturated with heptane and held with forceps. It was then rewiped in the same manner with the electronic grade precision cleaning agent (PCA) Freon. The next control test resulted in a very light condensate on the $-60^{\circ}C$ mirror. The source of this condensate was eventually identified as a methyl silicone virtually identical in its infrared spectrum to the Dow Corning high vacuum silicone grease used on the O-rings and the drive screw of the high vacuum gate valve. The measurements used to establish this are discussed in detail in Section 5.

The entire system was then disassembled and cleaned in a boiling caustic solution followed by heptane and Freon wipes. The 0-rings were put back dry and the drive screw of the high vacuum gate valve was coated with a MoS_2 base dry film lubricant. The chamber was then baked out and a control test was conducted. This control test with mirrors 481A and 481B had no visible deposit, therefore the first test was begun with 1/4-mil, one-side aluminized Mylar. During the first night the cold mirror platen experienced a thermal runaway when the solenoid failed, therefore, this control test was continued for one extra day to compensate for the possibility that any condensate formed before the thermal excursion might have been sublimed from the surface during the thermal excursion. The film formed during the Mylar test on the $-60^{o}C$ mirror was very light and one corner exhibited a slight interference fringe. The details of this test and subsequent tests and control runs are summarized in Table 4-1 and discussed in detail in Section 5. Because of the ultra-small amount of condensate visible on the $-60^{o}C$ mirror from the Mylar test, the next test series using RTV 60 silicone rubber was begun before any measurements were performed on the mirrors from the Mylar test series. RTV-60 produced a readily visible condensate on the $-60^{o}C$ mirror within a few hours. The temperature of the $+60^{o}C$ mirror was lowered in $10^{o}C$ increments when no condensate was visible after the first night of the test. Each temperature was maintained for at least four hours. It appeared that a very light film may have been forming at $+30^{o}C$. The "hot" mirror was held at that temperature for eight hours, without an appreciable increase in the visually observable film. When lowered to $+20^{o}C$ a readily visible film was formed. The temperature was not reduced below this point for the remainder of the environmental exposure. It should be noted that the films on the two mirrors visually appeared to be appreciably different in reflectance. During the second night of this test the solenoid for the cold mirror platen stuck and it experienced a high temperature excursion (greater than $300^{o}F$). During this time the condensed film on the cold platen changed from a glaring bright deposit to a cloudy one. It never again returned to its original appearance. The measurements performed on these mirrors are discussed in detail in Section 5.

The entire system was then solvent cleaned as described earlier and baked out over night at $+256^{o}F$. A short "control" test (one day) was then performed with mirrors 501A and 501B.

There was no visual deposit on these mirrors, therefore a standard control test was begun with mirrors 502A and 502B. The results of this control test were also visually good but as we discovered with 501A and 501B, these mirrors also exhibited a trace amount of hydrocarbon. This finding together with the Lyman α results are presented in Section 5. Four subsequent "control" tests performed with various parts of the system removed (e.g., Teflon sleeving, etc.) produced some surprising results; primarily, increase in the Lyman α reflectance due to environmental exposure and hydrocarbon contamination in the mirrors. These results are discussed in Section 5 together with other side experiments performed in an attempt to establish the reasons for what was observed. This included:

a. Infrared spectral measurements of many materials searching for the hydrocarbon source.

b. Exposing mirrors to thermal vacuum alone.

c. Extreme relative humidity exposure of the mirrors.

d. Various solvent cleaning techniques for the mirrors.

After the third "control" test, a standard control test was performed to obtain a reference for the next test material Sylgard 184, silicone rubber. The Sylgard 184 produced a heavy condensate on the $-60^{\circ}C$ mirror within a few hours. The warm mirror $+60^{\circ}C$ was lowered in $10^{\circ}C$ increments in a manner similar to that used in the RTV 60 test. This time the condensate appeared to start forming at $+20^{\circ}C$. The temperature was then lowered to $+10^{\circ}C$ at which time a very light film formed. When reduced to $0^{\circ}C$, the film remained about the same and did not appear to become thicker. The results of the measurements on these mirrors and some additional UV transmission measurements of a methyl silicone film are discussed in Section 5.

The chamber was cleaned as before and baked out, then a short "control" test was performed without measured slides. Since this did not produce any condensate, a standard control test was performed. The next material tested was Raychem Specification 44 Spacewire.

A very light film with interference fringes was noted on the -60°C mirror. The hot mirror was maintained at +60°C throughout this test. During this test a meeting was held at NASA Goddard to discuss the results obtained during the series of control tests before the Sylgard 184 test. It was agreed at this meeting by both the General Electric and the NASA personnel present that in order to conduct the program as originally conceived we would need stable mirrors, i.e., mirrors whose Lyman α reflectance did not change by more than $\pm$ 5% in a control test. The detailed experimental plan established at this meeting for the remainder of this program is given in Appendix A.

This consisted of conducting a control test at one half the normal ultraviolet intensity (5 EUVS rather than 10 EUVS). The purpose of this was to determine if the phenomenon observed was rate sensitive. After this we performed "control" tests with three pair of mirrors manufactured at NASA Goddard. Two pair were the standard size and one pair were 1/2-inch squares. The surface topography of these smaller mirrors was measured both before and after environmental exposure with an electron microscope at NASA Goddard; the only measurements performed on this particular set of mirrors. The purpose of the measurements was to determine if changes in the surface topography were responsible for the increase in Lyman α reflectance being observed. Both mirror platens in all three of these "control" tests were maintained at +60°C throughout the environmental exposure. This was done so that the maximum increase possible would be observed, if there was one. It can be noted from Table 4-1 that in general the Lyman α reflectance of the +60°C mirror increased both more frequently and to a greater extent than the -60°C mirror. The small 1/2-inch square mirrors and two of the standard size mirrors (one used in each of two "control" tests) were prepared on polished copper substrates. The other two standard size mirrors (one used in each of two "control" tests) were prepared on the standard ferrotype photographic plates that we were using throughout the program. These three "control" tests were to establish the following:

a. If stable mirrors were producible.

b. If the substrate was influential in obtaining a stable mirror.

c. If the surface topography was important in mirror stability.

The results of these tests are discussed in detail in Section 5.

Simultaneously with this modified experimental program a brief literature survey was conducted to determine what was already known about the stability of MgF_2 overcoated aluminum mirrors under near ultraviolet radiation. Other pertinent data on MgF_2 stability, such as, effect of deposition process, etc. was obtained as the search was conducted. The pertinent references and a brief description of the salient facts relevant to this problem are presented in Appendix B. A list of the materials investigated and a detailed description and identification of each is given in Appendix C.

Table 4-1. Summary of Measurements From Test Program

Mirror #	Type of Test	Temp., °C	Before Test	After Test	% Change	IR Analysis	Change in Mirror Weight (mg.)
481A	control	+60	.90	.68	-24	---	+ 0.3
481B	control	-60*	.63	.30	-52	Hydrocarbon	+ 2.3*
481C	none, control for 481A & B	----	.45	.66	+47	---	----
482A	Mylar	+60	.80	.97	+21	---	+ 0.1
482B	Mylar	-60	.94	.21	-78	mostly hydrocarbon and some methyl silicone	- 0.1
482C	none, control for 482A & B	----	.80	.85	+6		----
483A	RTV60	+60 (+20)	.90	.12	-87	mostly cross-linked methyl silicone & aliphatic hydrocarbon	+ 1.2
483B	RTV60	-60*	.81	.14	-83	(same as above)	+ 5.4*
483C	none, control for 483A & B	----	.88	.87	-1	---	----
501A	1-day control	+60	.08	.14, .14	+75	---	0.0
501B	1-day control	-60	.36	.57, .57	+58	trace of hydrocarbon	+ 0.1
502A	control	+60	.41, .43	.55	+28	trace of hydrocarbon	- 0.4
502B	control	-60	.50, .53	.33	-38	hydrocarbon and methyl silicone	- 0.4
512A	1-day control	+60	.51, .52	.58	+12	decreased carbon hydrogen absorption	+ 0.1
512B	1-day control	-60	.43, .40, .44	.49	+11	no significant change	+ 0.1
521C	3-day control	+60	.37, .38	.46	+21	no significant absorption	- 0.2
521D	3-day control	-60	.16, .15	.48	+220	hydrocarbon	- 0.3
513C	1-day control	+60	.50	.54	+8	no significant absorption	0.0
513D	1-day control	-60	.49	.42	-14	hydrocarbon	- 0.1
521C	control	+60	.46	.51, .53	+11	no significant absorption	+ 0.1
511A	control	-60	.38	.36, .37	-5	hydrocarbon	+ 0.1
521C	Sylgard 184	+60 (+10)	.51, .53	.48	-9	---	0.0
541D	Sylgard 184	-60*	.39	.36	-8	methyl silicone	+ 5.5*
512C	control	+60	.34, .34, .42	.53	+26	---	0.0
512B	control	-60	.42, .45	.43	4	---	0.0
521A	Spec. 44 Raychem Space Wire	+60	.42, .43	.54	+25	nothing detected	- 0.2
521B	Spec. 44 Raychem Space Wire	-60	.39, .37	.06	-84	hydrocarbon and silicone	+ 0.3

( ) indicates temperature at which condensate formation became visible

* a thermal excursion to a temperature greater than 300° F was experienced by this mirror during its environmental exposure.

SECTION 5

DISCUSSION OF RESULTS

5.1 CHECKOUT OF EQUIPMENT AND EXPERIMENTAL TECHNIQUES

The Lyman α reflectance of the test mirrors was measured relative to the reflectance of a reference mirror taken as 1.00. An initial series of reflectance measurements was made with a set of three mirrors designated 1A, 1B, and 1C. These measurements were made to check the reproducibility of the technique utilized. All mirrors were coded with a numeral and a letter. The letter designates the position of the mirror in the measurement device (i.e., A represents the top position, B the second, C the third), while the numeral represents the set of measurements. Thus, mirror 3B was one of the third group of mirrors to be measured and was placed in the second position on the measurement device.

The relative reflectance of the mirrors of set 1 was determined four times. The measurement was made the first thing in the morning. The mirrors were removed and other sets measured during the day. Set 1 was then returned to the vacuum chamber and the measurement repeated. These mirrors were left in the chamber overnight and the measurement repeated on the following morning. This set of mirrors was then removed from the chamber and other measurements were made during the day. Finally, set 1 was returned to the chamber and the fourth measurement of set 1 was made on the second afternoon. It is obvious from the data presented in Table 5-1 that the mirrors, having been sheared and punched rather than machined, were not flat enough to be used in the reflectance apparatus as originally designed. Therefore, the mirror holder was redesigned so that the mirror was held by bolts at all four corners. This ensured that the mirror would be as flat as possible during measurement and would be placed in very nearly the identical position during all reflectance measurements.

In the early part of the program a number of control runs were made which resulted in the collection of visible condensate on the mirrors. Infrared analysis of this condensate clearly indicated the presence of aromatic ester with long-chain aliphatic groups (See Figure 5-1). Comparison with a Sadtler standard spectrum and with a spectrum of diallyl phthalate

Table 5-1. Initial Lyman - α Reflectance

Mirror Number \ Day Measurement	Relative Lyman - α Reflectance 1 1	1 2*	2 1	2 2*
1A	0.81	0.85	0.84	0.78
1B	0.74	0.78	0.78	0.74
1C	0.98	0.98	0.98	0.95

*Indicates mirror removed and replaced before 2nd measurement.

(Figure 5-2) confirmed this analysis. An intensive search for the source of this contamination included analysis of the mechanical pump oil (Figure 5-3), the diffusion pump fluid (Figure 5-4), extracts from an electrical connector (Figure 5-5) and the neoprene bell jar gasket (Figure 5-6). Finally as was mentioned in Section 4, it was learned that poly (vinyl chloride) gloves were being used during solvent cleaning of the vacuum system. The cleaning agent, Freon PCA, is very effective at extracting the phthalate plasticizer from the poly (vinyl chloride) gloves. The infrared spectrum of this extract is shown in Figure 5-7.

Freon extracts of other types of gloves were analyzed to determine the suitability of such gloves for use in cleaning the vacuum system. So-called rubber gloves (Fisher Scientific Co.) yielded a large amount of extract. The resultant spectrum was complex and indicates the probable presence of an aromatic ester, hydroxyl groups, and carbon-oxygen bonds other than those found in the ester. It is probably a mixture of several components. A similar extraction of "Compar Plastic"† gloves yielded only a very small amount of a phthalate ester. As can be seen in Figure 5-9 the infrared analysis of this extract was run at 5X and 10X scale expansion so the amount of extracted material was very small. Extraction of a Nylon glove and a polyethylene glove also gave very small amounts of material. Infrared

† T. M. The Wilson Rubber Co.

analysis of the former extract indicated that it was probably a mixture of aliphatic acid and ester (Figure 5-10) while analysis of the latter extract indicated aliphatic hydrocarbon (Figure 5-11).

Once the major contaminant (phthalate ester) had been eliminated from the vacuum chamber, further control runs were performed. A much lower level of contamination was observed in these runs. The material appeared to be largely aliphatic hydrocarbon and silicone (Figure 5-12). These contaminants were prabably present all along but were masked by the much greater amount of phthalate present earlier. As was discussed in Section 4 the contamination was further reduced by removal of all silicone grease from the system.

A subsequent control run resulted in decreases in reflectance of both hot and cold mirrors, 481A and 481B respectively. A companion mirror, 481C, was measured along with 481A and 481B though it was not placed in the vacuum chamber. An inordinately large increase was observed in the reflectance of this mirror. The change was far beyond our normal error limits, and we are unable to explain it.

5.3 TEST MEASUREMENTS

The measurements performed during the testing phase of this program are summarized in Table 4-1 and 5-2. This includes Lyman α reflectance, FMIR infrared measurements of the mirrors (both before and after environmental exposure), and the change in weight of both the mirror and the sample due to environmental exposure (as determined from before and after weighings).

Table 5-2. Bulk Weight Loss

Material	Sample Weight (grams)	Dimensions (inches)	Weight Loss (per cent)
RTV 60	5.8954	2.3 x 2.0 x .059	0.49
Sylgard 184	5.4762	2.3 x 2.0 x .077	0.44
NRC-2	0.3380	8.5 (diameter) x $2.5 \cdot 10^{-4}$	0.00
Spec. 44 Spacewire	3.8479	12 (length)	0.00

The reflectance at Lyman α of three aluminum mirrors bearing the designations 482A, 482B, and 482C was measured. Values (relative to the standard) of 0.80, 0.94, and 0.80, respectively, were obtained. These mirrors were then used in an experiment with Mylar as a possible source of contamination. Reflectance of the mirrors was measured after environmental exposure and values of 0.97, 0.21, and 0.85 were obtained. Mirror 482C was not exposed in the vacuum system but served as sort of a secondary standard. Mirror 482B lost 78% of its reflectance at Lyman α, as well as a substantial (though measured) portion of its reflectance in the visible. An increase in reflectance of 482A was observed. This change was well beyond our normal experimental error. At first it was thought that the apparent increase might actually represent a decrease in reflectance of our "standard" mirror. But this is unlikely since subsequent cleaning of the "standard" mirror with Freon PCA had no effect on its reflectance; and the reflectance of the "standard" relative to that of a reference mirror which was kept under nitrogen was substantially the same at this time as it was at the beginning of the program.

The contaminant coating on 482B was analyzed by infrared spectroscopy using FMIR and 5X scale expansion. Absorption maxima were observed at 2910, 2850, 1440, 1365, 1259, and 1180-1120 cm^{-1}. The mirror was then washed with carbon tetrachloride and the solvent evaporated on a salt plate. The residue had absorption maxima at 2940, 2910, 2850, and 1460 cm^{-1}. These facts can be interpreted in the following way:

Mirror 482B was contaminated with a mixture of at least two materials. One was a aliphatic hydrocarbon, relatively high molecular weight and with little branching. The other was a methyl silicone which had been cross-linked by ultraviolet light and was insoluble in carbon tetrachloride and methylene chloride. Neither of these contaminants is native to pure Mylar, nor is it obvious how they could be produced from decomposition products.

The reflectance at Lyman α of mirrors 483A, 483B, and 483C was measured, and values of 0.90, 0.81, and 0.88, respectively, were obtained. These same mirrors were measured a second time after being exposed in an experiment with RTV-60. At that time values of 0.12, 0.14, and 0.87 were obtained. Mirrors 482A and B both had an obvious contaminant coating, so the reflectance values of 0.12 and 0.14 were surprisingly high. Infrared

analysis showed that the coating on 483B was a methyl silicone. It was cross-linked and insoluble in carbon tetrachloride but did wrinkle and come off the mirror when immersed in carbon tetrachloride. The coating on 483A also proved to be a methyl silicone, but this coating was apparently unaffected by carbon tetrachloride. Washing 483A in carbon tetrachloride did, however, remove an estimated 0.2 mg. of an aliphatic ketone.

Mirrors 501A and 501B were measured after a one-day control run. Reflectance values were 0.14 for 501A and 0.57 for 501B. These were significantly higher values than the 0.08 and 0.36 (respectively) that were obtained originally. It seemed possible that this increase in reflectance might be due to removal of moisture from the magnesium fluoride overcoat, so 501B was kept overnight at 35°C, 40% R.H., while 501A was kept in a desiccator over a silica gel. Reflectance measurements made the following day gave identical results, i.e., 0.14 and 0.57, indicating that moisture is not likely to be a factor in these measurements. An FMIR spectrum of 501B clearly showed the presence of carbon-hydrogen bonds but little else.

The reflectance of mirrors 502A and 502B was measured after a four-day control run. Values of 0.55 and 0.33, respectively, were obtained. Thus, mirror 502A increased in reflectance from its original value of 0.41 while 502B decreased from its original value of 0.50. An FMIR spectrum of 502A showed the presence of a small amount of hydrocarbon, while a spectrum of 502B showed the presence of both a hydrocarbon and a methyl silicone.

Mirrors 521A, 521B, 521C, and 521D were ultrasonically cleaned in Freon ^{D}CA. Lyman α reflectance values before and after cleaning were as follows:

	Before	After
521A	0.42	0.43
521B	0.39	0.37
521C	0.37	0.38
521D	0.16	0.15

Mirrors 512A, 512B, 512C, and 512D were washed with heptane and Freon PCA. Reflectance values before and after washing were as follows:

	Before	After
512A	0.51	0.52
512B	0.42	0.45
512C	0.34	0.42
512D	0.40	0.44

Mirrors 512A and D were subsequently exposed in an overnight control run. Reflectance values of 0.58 and 0.49, respectively, were obtained. It was at this point that some peculiar data were obtained. Before the control run, mirror 512A had shown a relatively strong carbon-hydrogen absorption, but this absorption had nearly disappeared after the control run. It is therefore tempting to attribute the increased reflectance of 512A to clean-up in the vacuum. However, 512D also showed an increase in reflectance after the control run, but the relatively low carbon-hydrogen absorption of this mirror did not change appreciably. Irradiation at high temperature in vacuum appears to contribute to improvement of some mirrors and may account for observed increases in reflectance (see Appendix B). Mirrors 521C and 521D were exposed in a control run, after which a relatively large amount of hydrocarbon was found by FMIR, especially on 521D. The remarkable thing, though, was the increase in reflectance of 521D from 0.15 to 0.48.

In an attempt to improve their reflectance, six mirrors were heated at 120°C in vacuum. Reflectance values before and after heating were:

		Reflectance	
Mirrors	Heating Hrs.	Before	After
511B	3	0.30	0.38
511D	3	0.23	0.25
511A	20	0.41	0.38
511C	20	0.34	0.33
513C	17	0.50	0.50
513D	17	0.51	0.49

Except for 511B, heating in vacuum did not improve the reflectance of any of these mirrors by a significant amount.

One explanation for the observed results which seemed attractive at first was the possibility that hydrocarbon contaminants on the exposed mirrors fluoresced and gave a misleading impression of high reflectivity. If this were so, the fluorescence would most probably be at wavelengths longer than ~ 1600Å and would probably occur at wavelengths ≥ 2000A. The detector used for reflected Lyman α was sodium salicylate on a quartz window. When this window was reversed so that the sodium salicylate faced away from the mirror, no signal was detected from either the reference or mirror 521D. This proved that any light coming from these mirrors (when they were illuminated at Lyman α) is of a wavelength shorter than 2000 Å. A detector consisting of sodium salicylate on calcium fluoride was substituted. Reversal of this window decreased the signal from the reference mirror to 34% of its normal value, and decreased the signal from mirror 521D to 36% of its normal value. The reflectance of 521D relative to the reference was 0.49 compared to 0.48 with the quartz window. This shows beyond any reasonable doubt that the spectral distribution of light from the reference mirror and from 521D was the same--in other words, 521D had been cleaned by exposure in vacuum and was not fluorescing.

Despite the evidence described above, we continued the investigation of the possible fluorescence of contaminated mirrors. In our normal experimental arrangement, the mirror was located at the exit slit of the monochromator. Light reflected from the mirror was detected by sodium salicylate on quartz. However, this arrangement gives no direct measure of the spectrum of light coming from the mirror. Therefore the apparatus was altered so that the mirror was at the entrance slit and was illuminated with the total lamp output. With this arrangement the spectral distribution of light coming from the mirror could be measured. The spectrum from the reference mirror was recorded from 1200Å to 2000Å. Lines were evident at 1216Å, 1512Å, 1545Å, 1560Å, 1600Å, 1669Å, 1790Å, and 1925Å. Similarly the spectrum of light from mirror 541D was recorded after exposure of this mirror to products from Sylgard 184. The mirror was coated with an obvious layer of methyl silicone but lines were observed at 1216Å, 1545Å, 1600Å, and 1640Å. The intensities

of these lines relative to those of the reference mirror were 0.33, 0.12, 0.12, and 0.12, respectively. The value of .33 at Lyman α was in fairly good agreement with a measurement of .36 made before the change in experimental arrangement. It was thus quite clear that the high reflectance values obtained in the presence of silicone contamination were real and were not artifacts due to fluorescence. But it was still possible that the high reflectance was due to unusually high front-surface reflection from the silicone rather than transmittance of the silicone and reflectance of the aluminum. Therefore the transmittance of a piece of polished lithium fluoride was measured at Lyman α. A layer of silicone fluid SF96 painted on the lithium fluoride with a cotton swab reduced the percent transmission from 49 to 23.4. The transmission of SF96 was measured in this fashion with the following results:

λ, Å	% T
1725	<25
1650	20
1600	15
1550	8
1216	39

Although we do not know the thickness of the SF96 silicone layer in this experiment, it was thick enough to have some tendency to run when the surface was kept vertical for several hours. It was thicker than any deposit seen in this work with the exception of mirrors exposed to Sylgard 184 or RTV-60, and was comparable to these deposits. It is obvious, then, that polydimethylsiloxane is remarkably transparent at Lyman α and the high reflectances measured in the presence of this deposit are real. It should be noted that, although the amount of condensate on the mirrors in the Sylgard 184 test and RTV-60 test appeared to be the same both visually and from their increase in weight, the reduction in Lyman α reflectance caused by the RTV-60 condensate was an order of magnitude greater than that caused by the Sylgard 184 condensate. The only known difference in the two condensates is that from the RTV-60 was polymerized whereas that from the Sylgard 184 was not. It is possible that this difference caused at least the major amount of difference noted in the Lyman α reflectance degradation of the mirrors contaminated with these two condensates.

Table 5-3. Summary of Measurements From Modified Section of Program

Mirror Number	Type of Test	Temperature (°C)	Lyman Reflection			FMIR Analysis	Change in Mirror Weight (mg)
			Before Test	After Test	Per cent Change		
571A	control +	+60	0.29	0.43	+48	trace of Silicone and hydrocarbon	0.0
571B	control +	-60	0.59	0.60	+ 2	hydrocarbon trace	+0.2
581C*	control	+60	1.66	1.56	- 6	nothing detected	+0.1
581A**	control	+60***	1.61	.94	-42	nothing detected	+0.6***
581D*	control	+60	1.81	1.73	- 4	nothing detected	0.0
581B**	control	+60	1.69	1.75	+ 3	nothing detected	-0.1

+ These control tests were conducted at 5EUVS all others were run at 10 EUVS; all control tests were run for nominally 1000 EUVSH

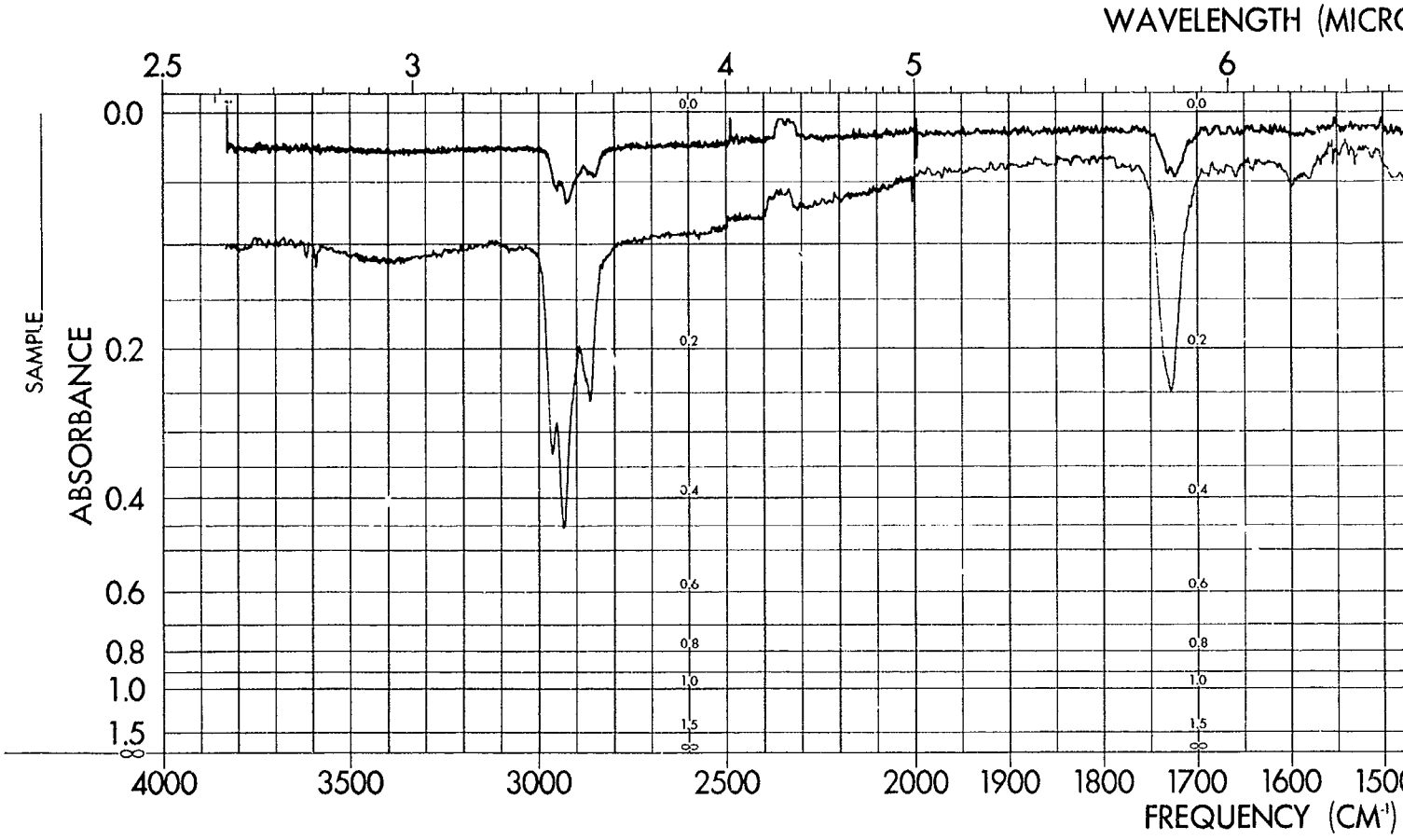
* Mirror prepared on a ferrotype plate substrate by NASA, Goddard

** Mirror prepared on a polished copper substrate by NASA, Goddard

*** A thermal excursion to a temperature greater than 300°F was experienced by this mirror during its environmental exposure.

Finally, two control runs were made with standard size mirrors prepared at NASA, Goddard. These mirrors had reflectance values about 1.7 to 1.8 times as great as those of the best of the other mirrors used earlier in this program. The measurements performed on these mirrors and those used for the 5 EUVS environmental exposure are summarized in Table 5-3. It is believed that the decrease in Lyman α reflectance of mirror 581A is related to the temperature excursion which it experienced during environmental exposure. It is possible that this is due to interdiffusion of the aluminum and the copper substrate. In no case was an increase in reflectance of the Goddard mirrors observed which was outside of experimental error; therefore, the increases observed throughout this program are characteristic of mirrors prepared in one fashion, but are not characteristic of magnesium fluoride overcoated aluminum mirrors in general, and do not appear to be substrate dependent. One obvious difference between the mirrors prepared at Goddard and those purchased for use on this program is the thickness of the magnesium fluoride coating. The former are coated with about 300Å of magnesium fluoride while the purchased mirrors had about a 1500Å layer. This is one of the reasons for the higher Lyman α reflectance noted on the Goddard mirrors compared to those from Evaporated Coatings Incorporated. The reflectance of the Goddard mirrors are about 70 percent more reflective than those produced by Evaporated Coatings Incorporated (independent of substrate).

FOLDOUT FRAME 1



FOLDOUT FRAME 2

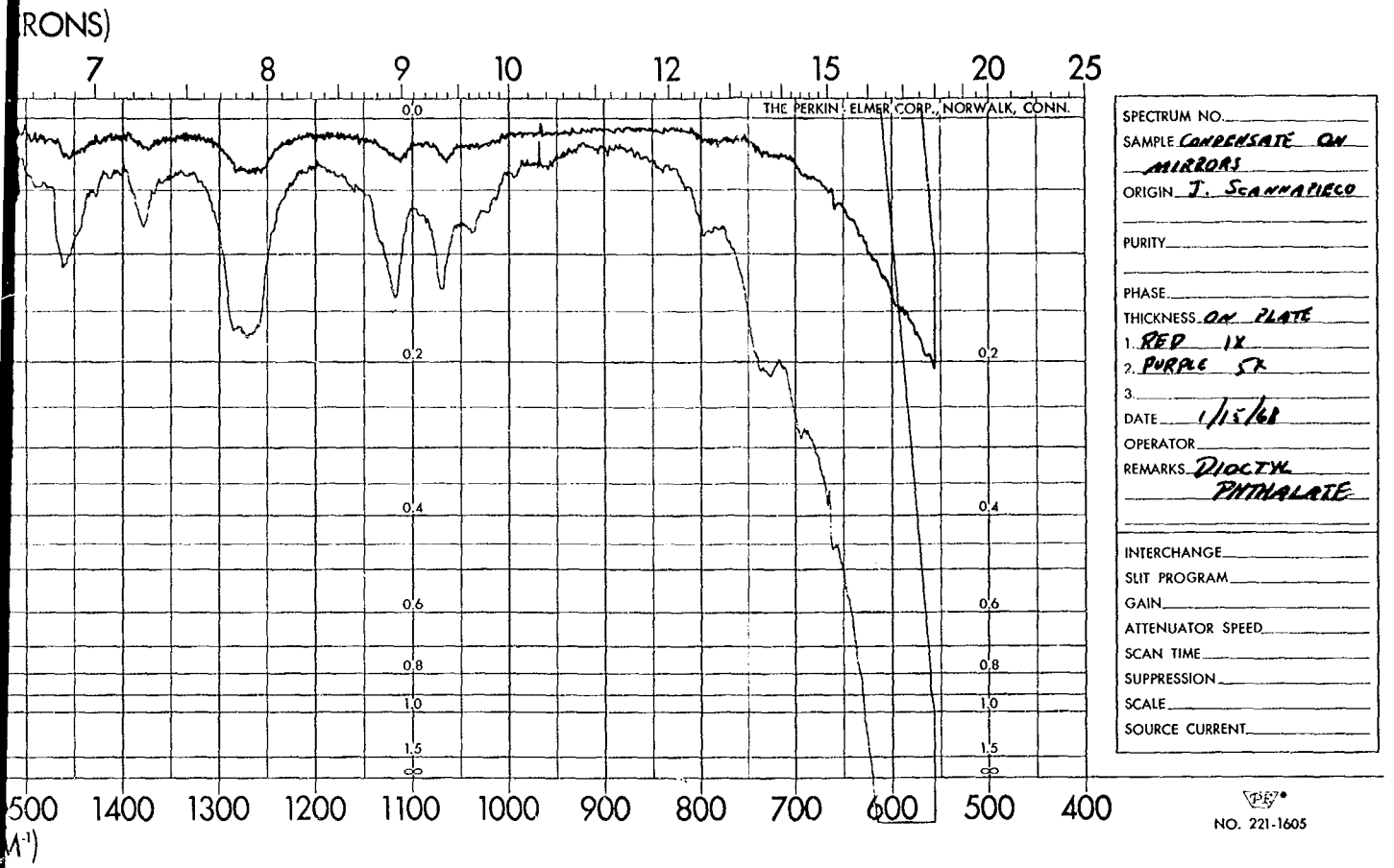
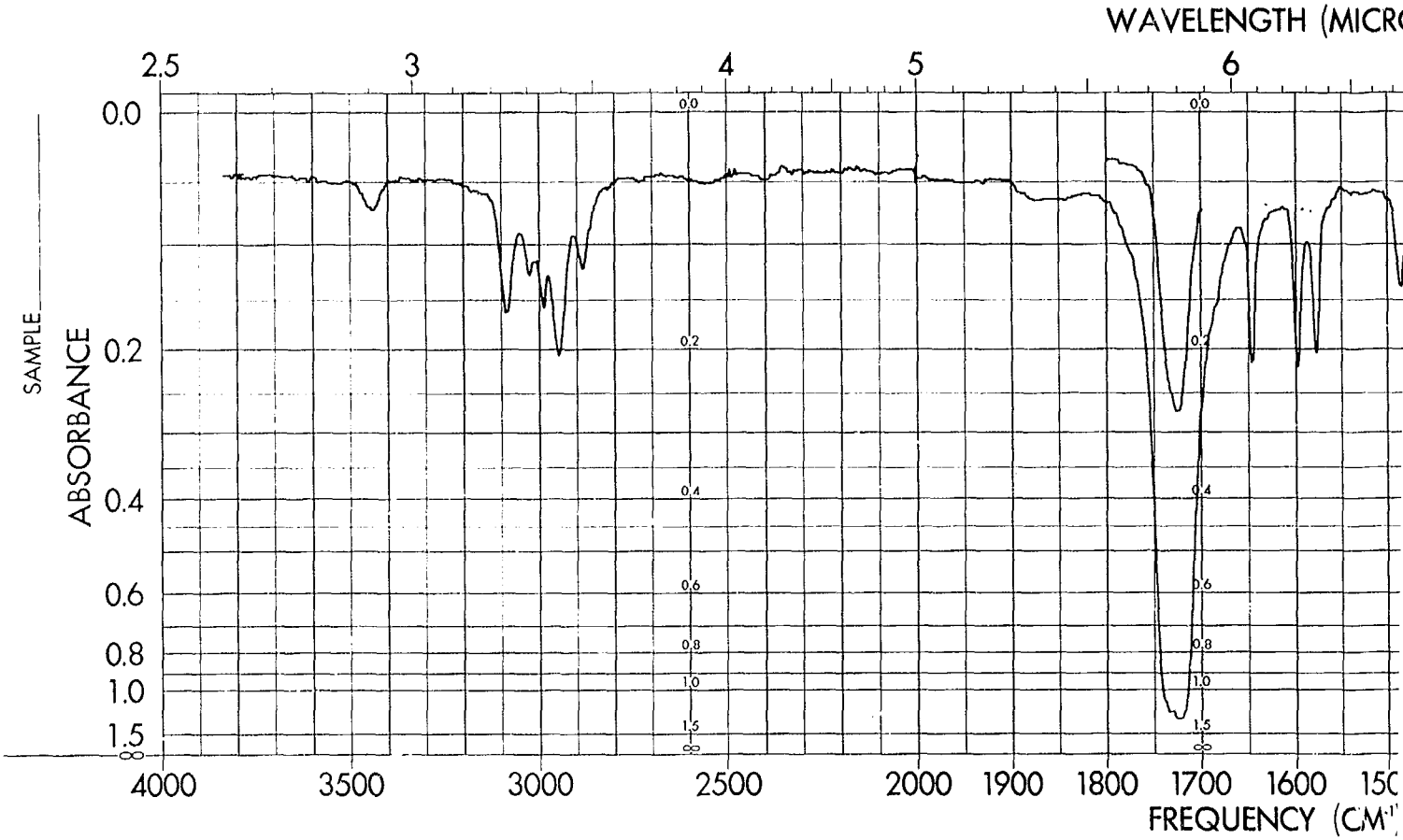


Figure 5-1. Initial Mirror Condensate


WAVELENGTH (MICR
2.5
3
4
5
6
SAMPLE
ABSORBANCE
0.0
0.2
0.4
0.6
0.8
1.0
1.5
∞
4000
3500
3000
2500
2000
1900
1800
1700
1600
150
FREQUENCY (CM⁻¹)

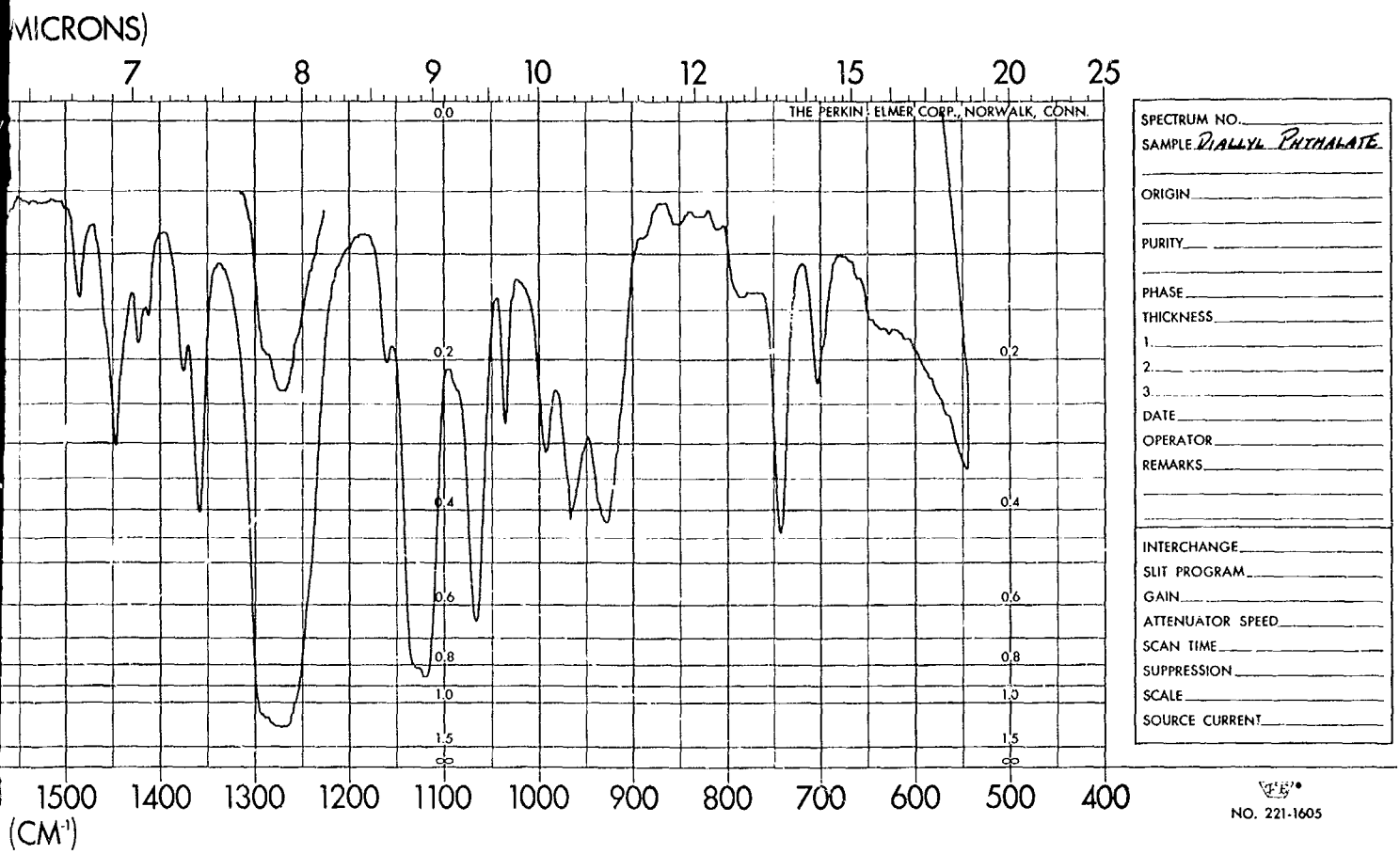
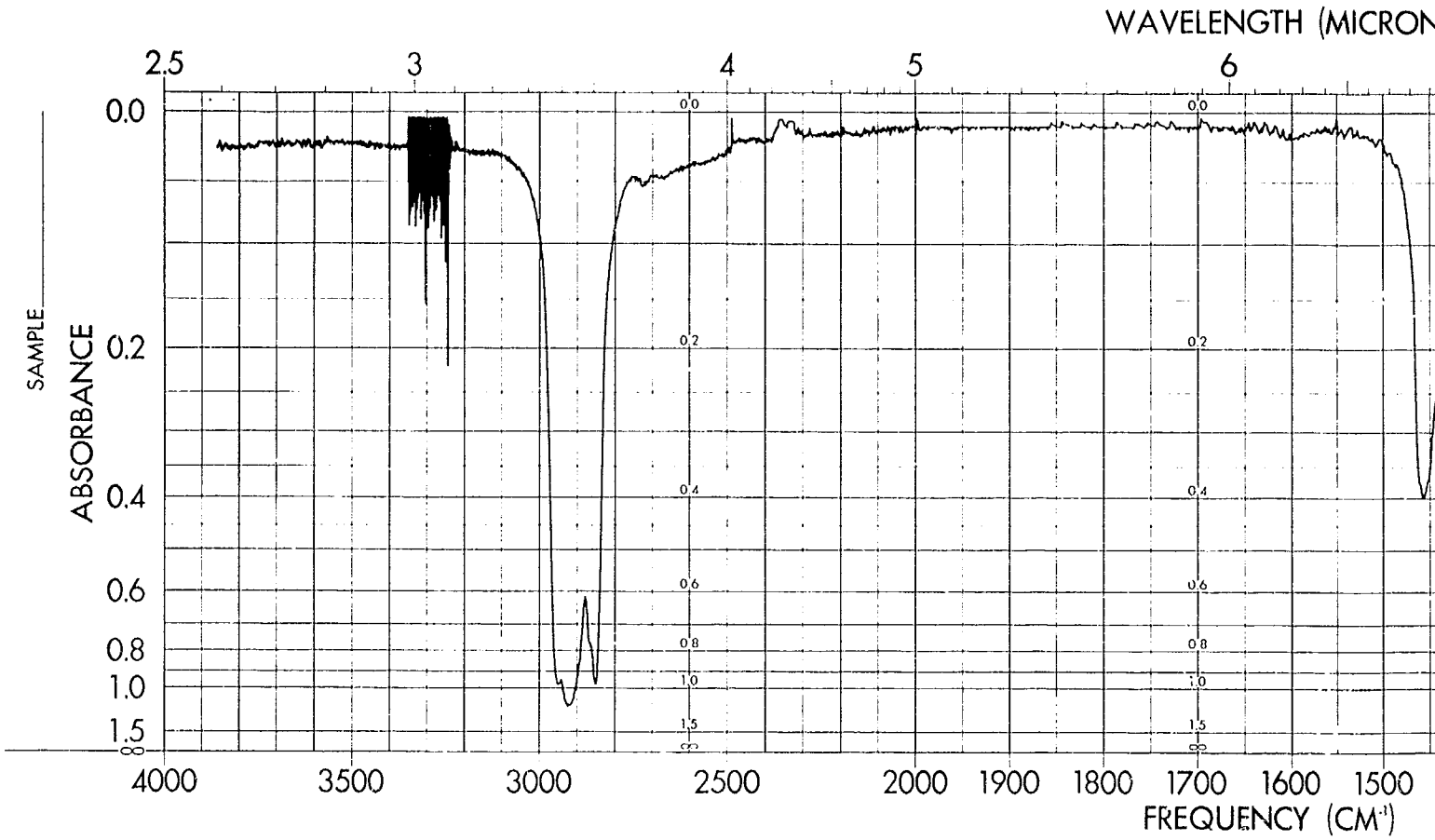


Figure 5-2. Diallyl Phthalate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FREQUENCY (CM-1)

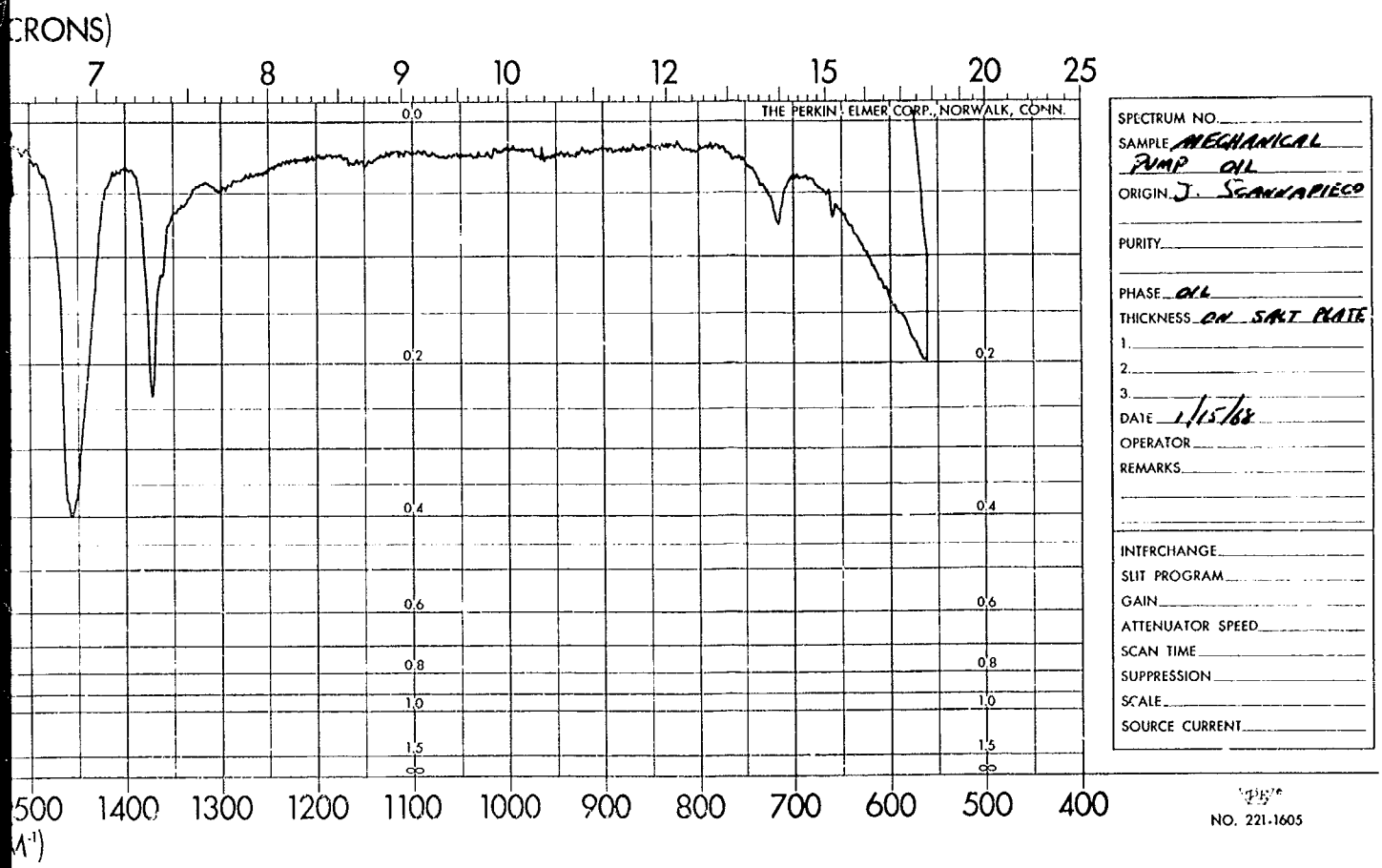
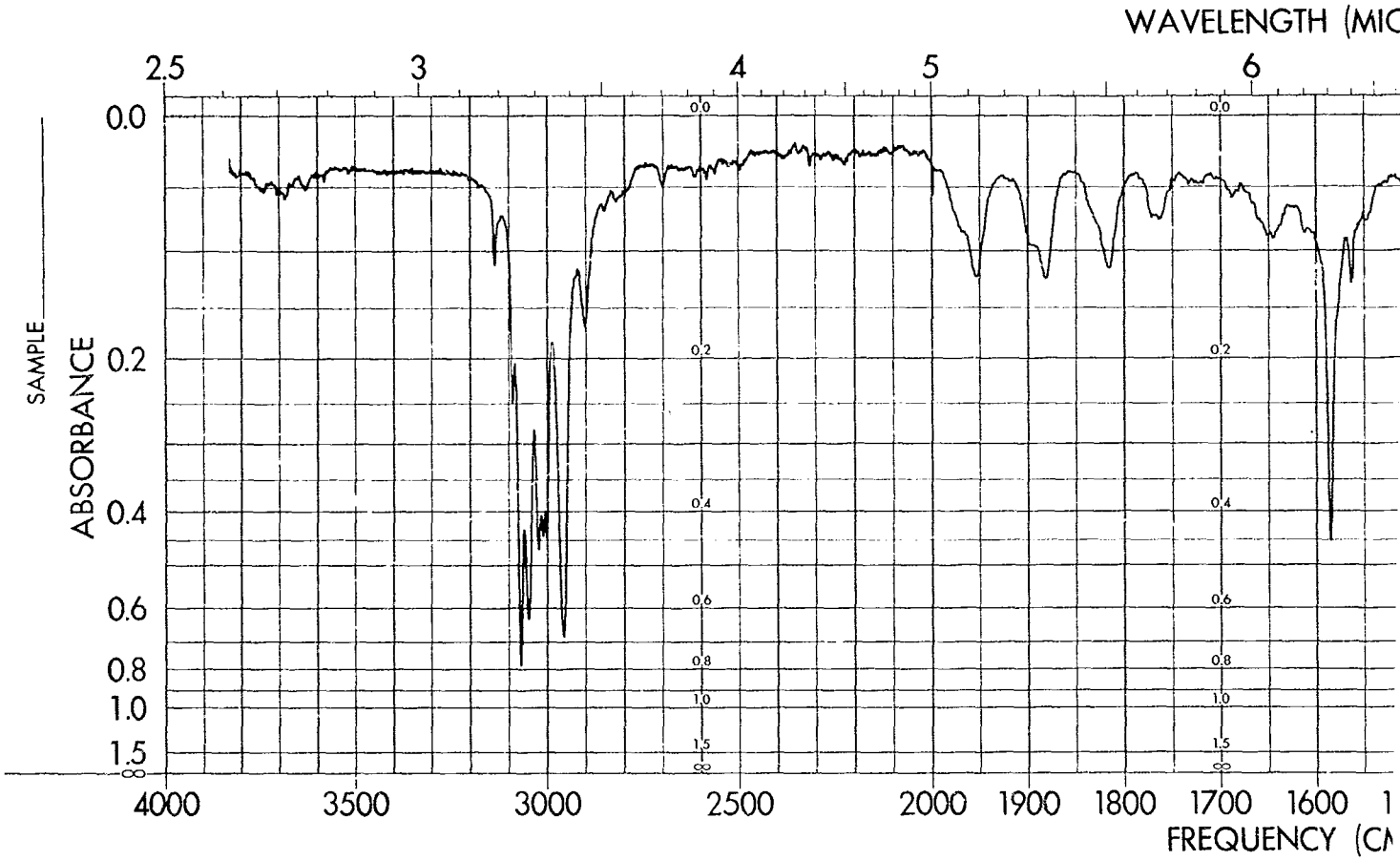


Figure 5-3. Welsh Duo Seal (number 17) Mechanical Pump Fluid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FREQUENCY (CM

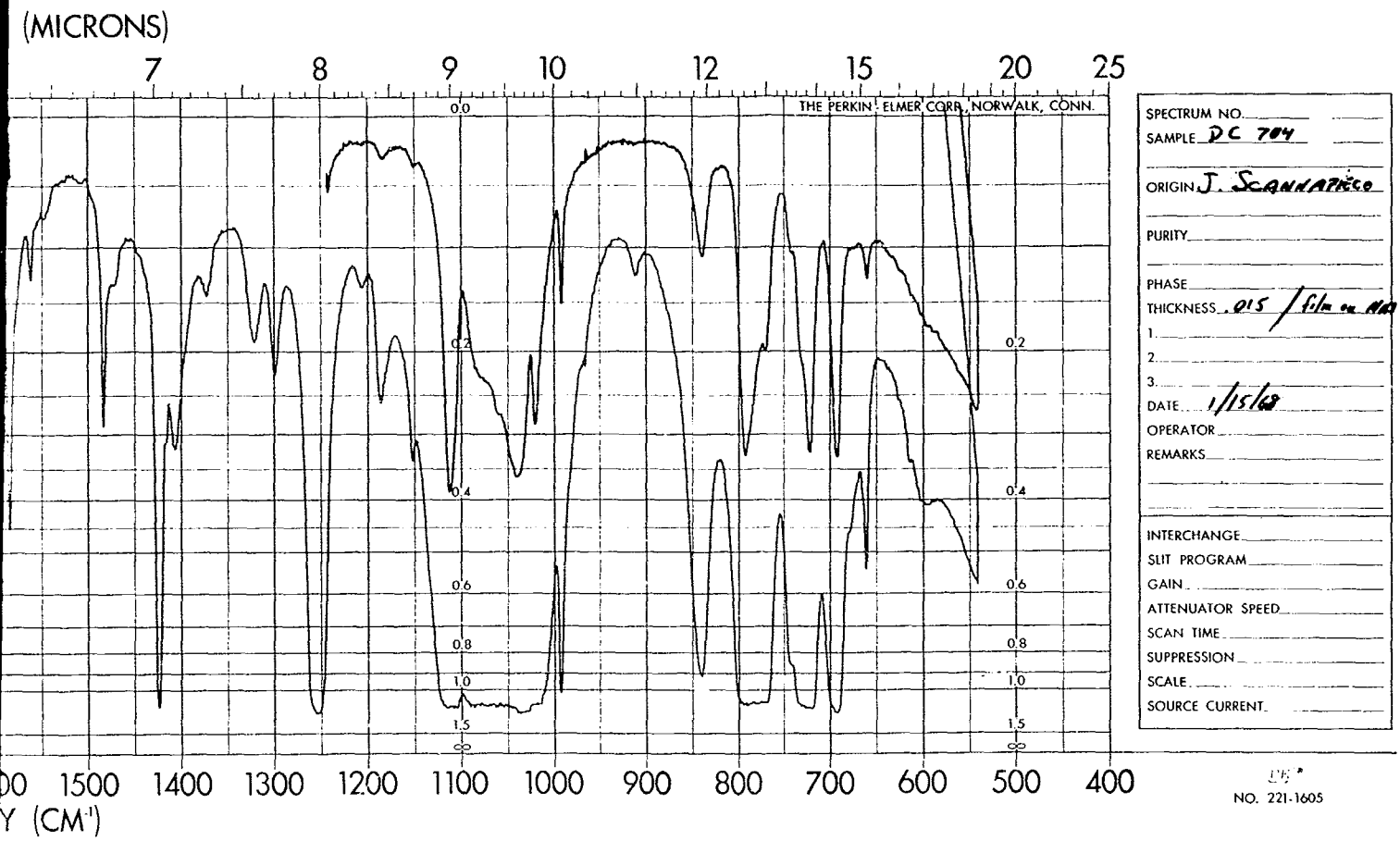
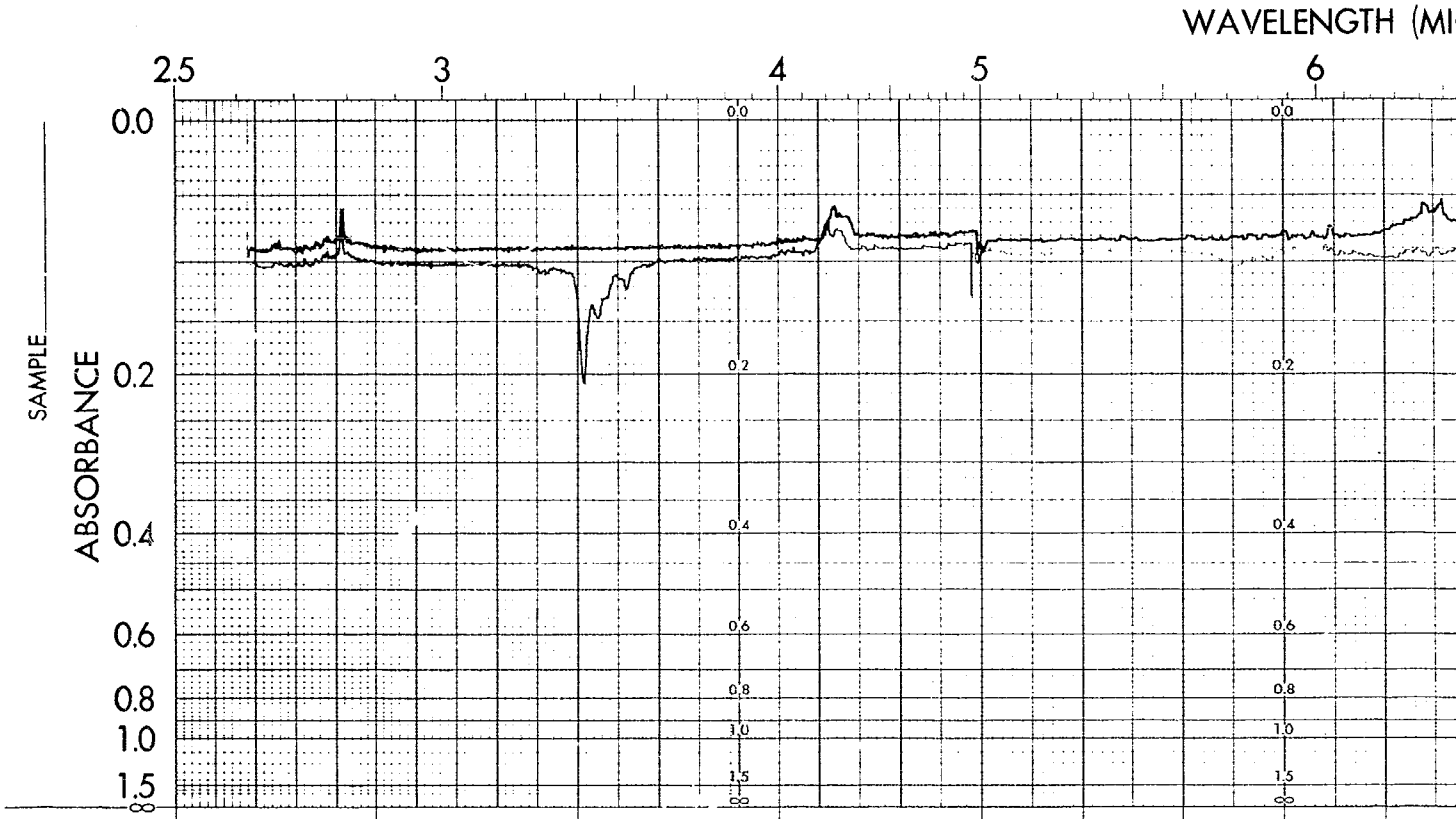


Figure 5-4. DC 704 Diffusion Pump Fluid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SAMPLE
ABSORBANCE
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FREQUENCY (C

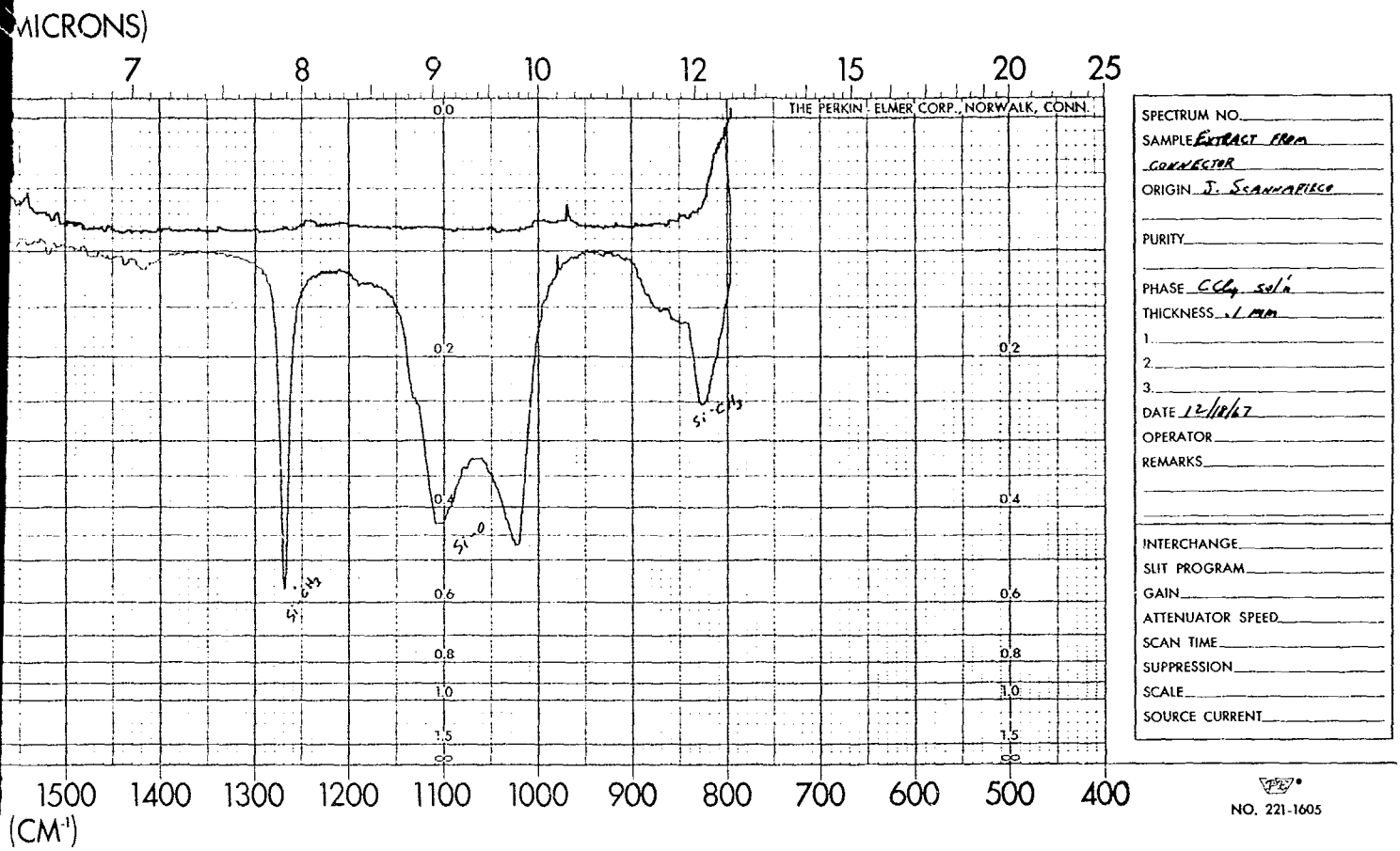
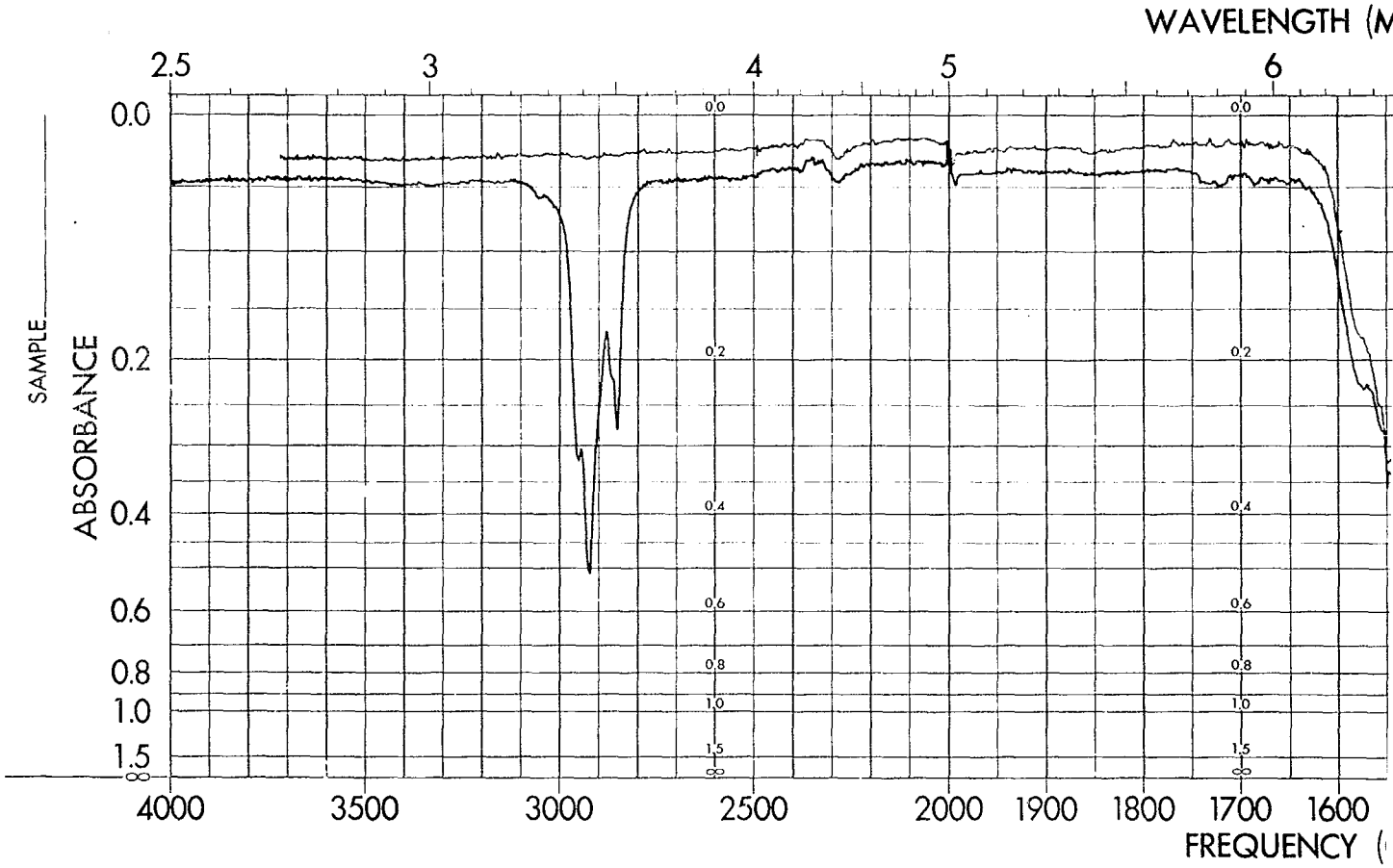


Figure 5-5. Extract From Deutsch Electrical Connector


WAVELENGTH (M
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ABSORBANCE
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FREQUENCY

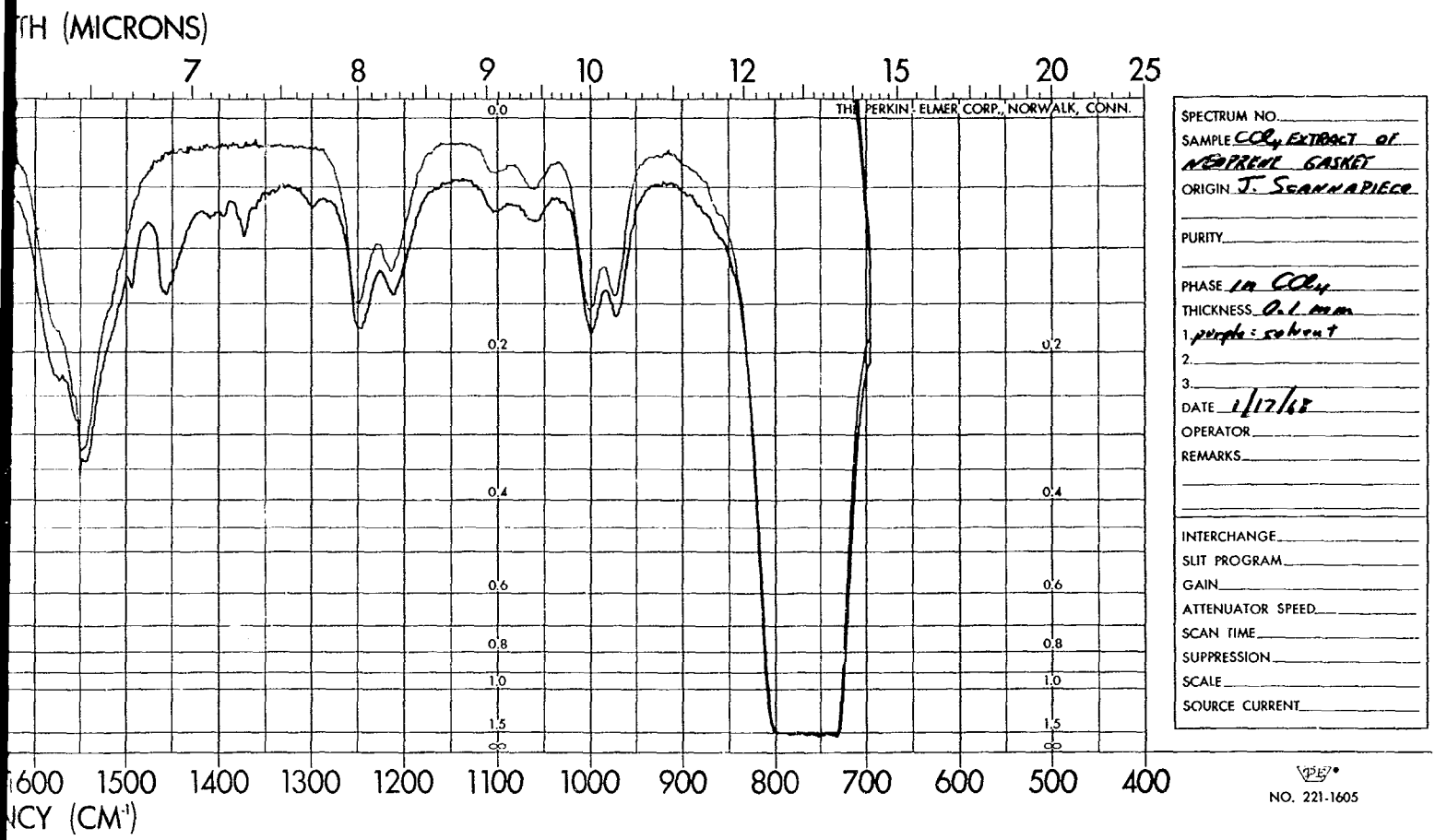
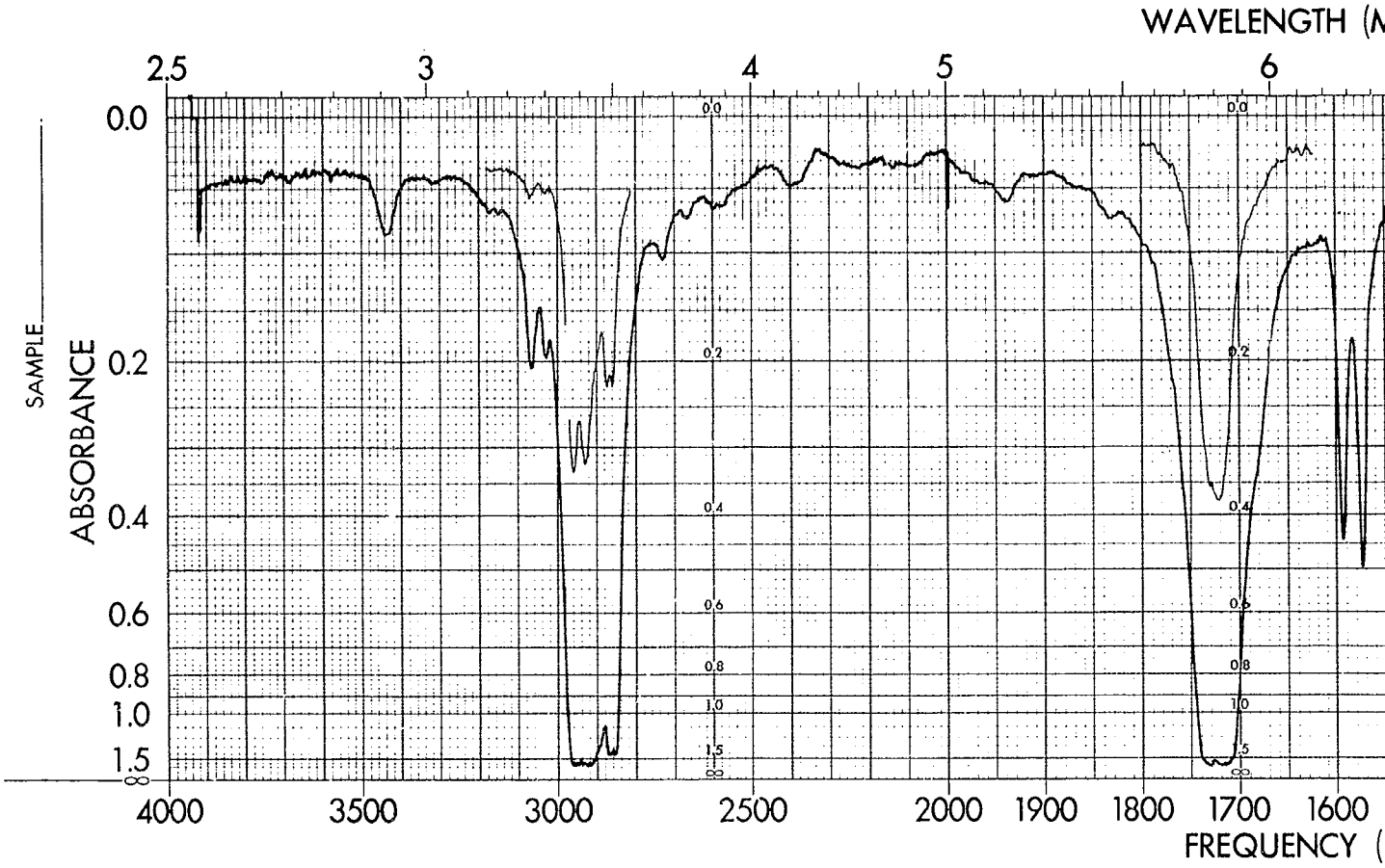


Figure 5-6. Extract From Neoprene Bell Jar Gasket


WAVELENGTH (M
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SAMPLE
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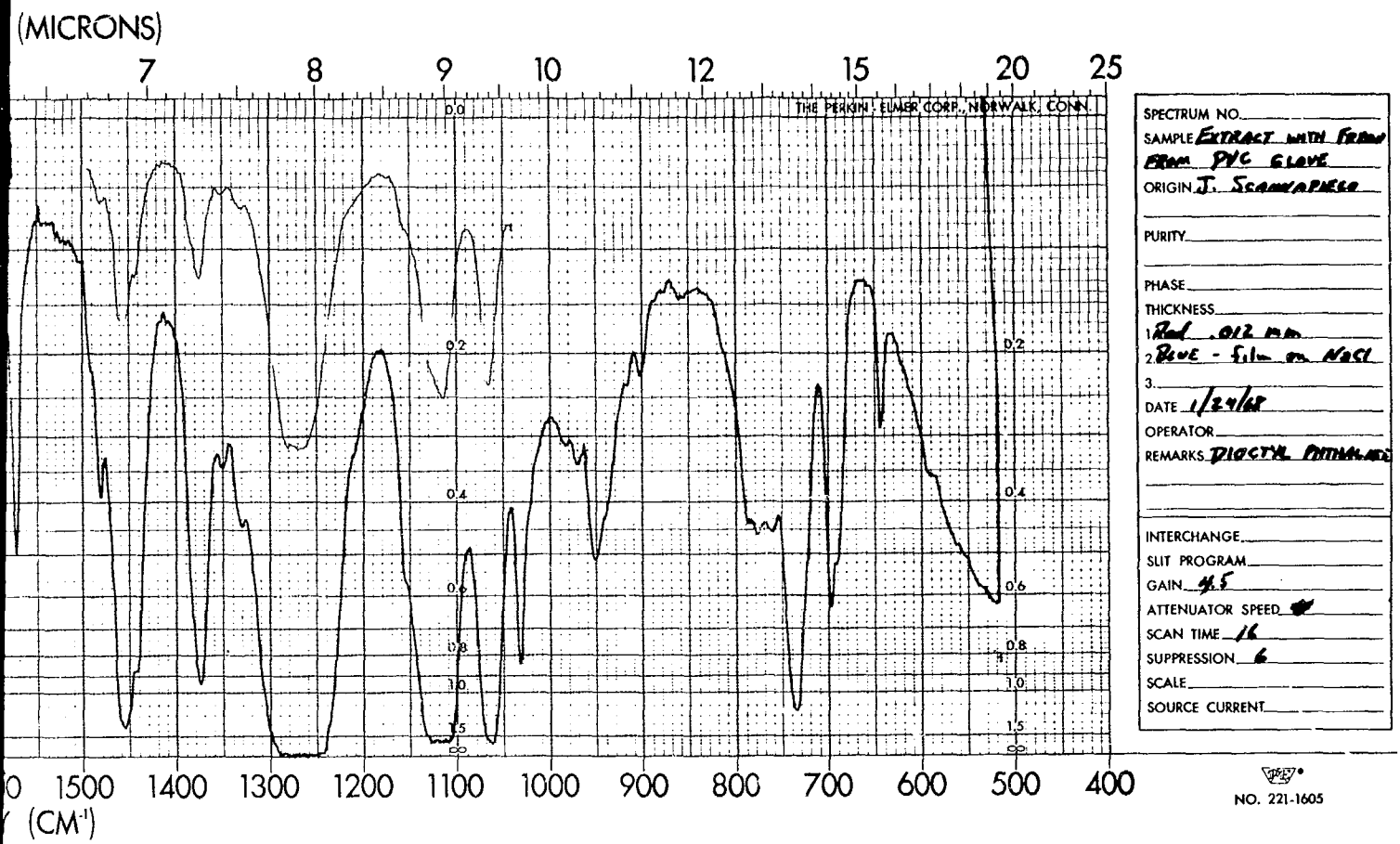
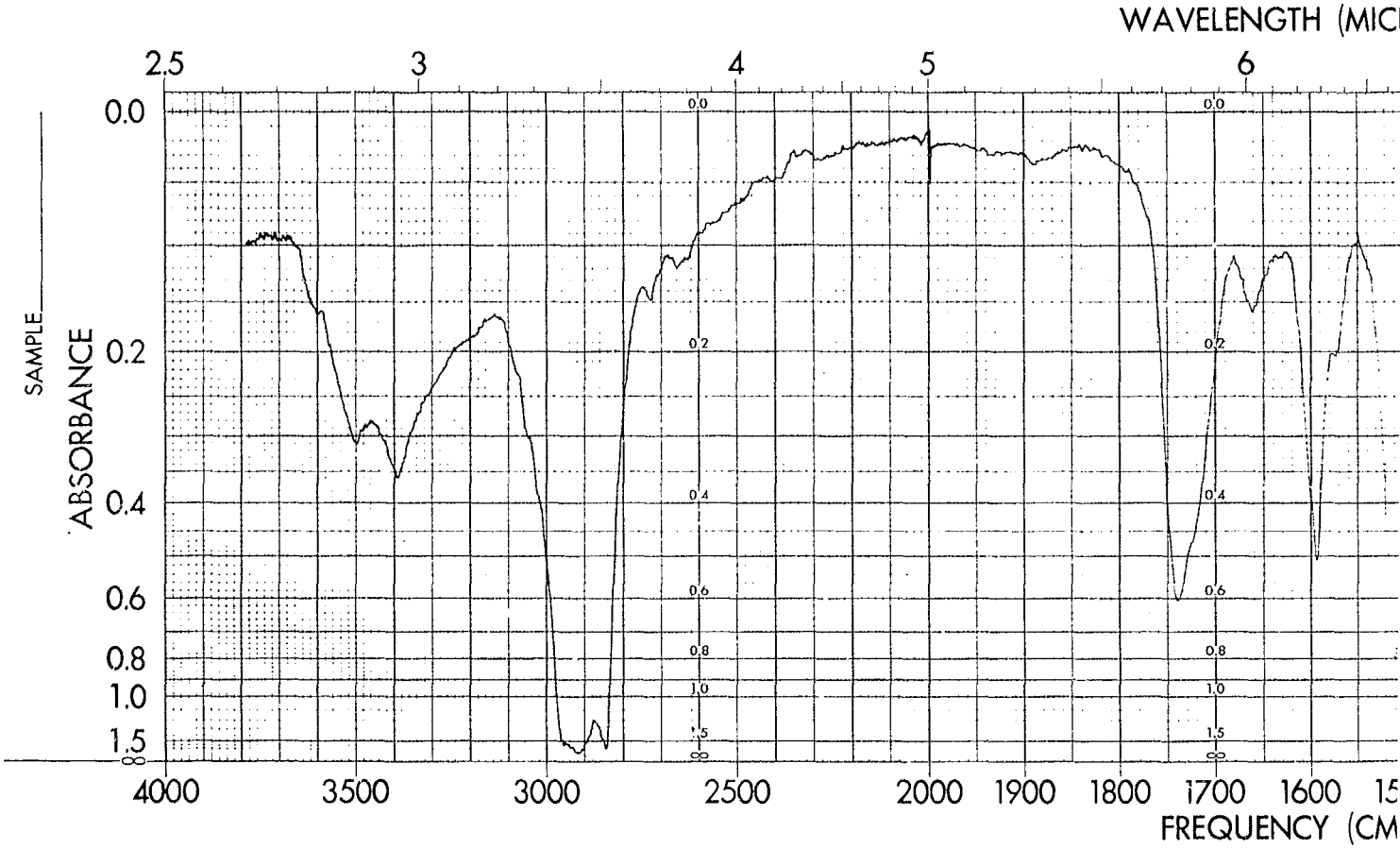


Figure 5-7. Poly Glove Extract


WAVELENGTH (MIC
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6
SAMPLE
ABSORBANCE
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FREQUENCY (CM

EXAMPLE 2

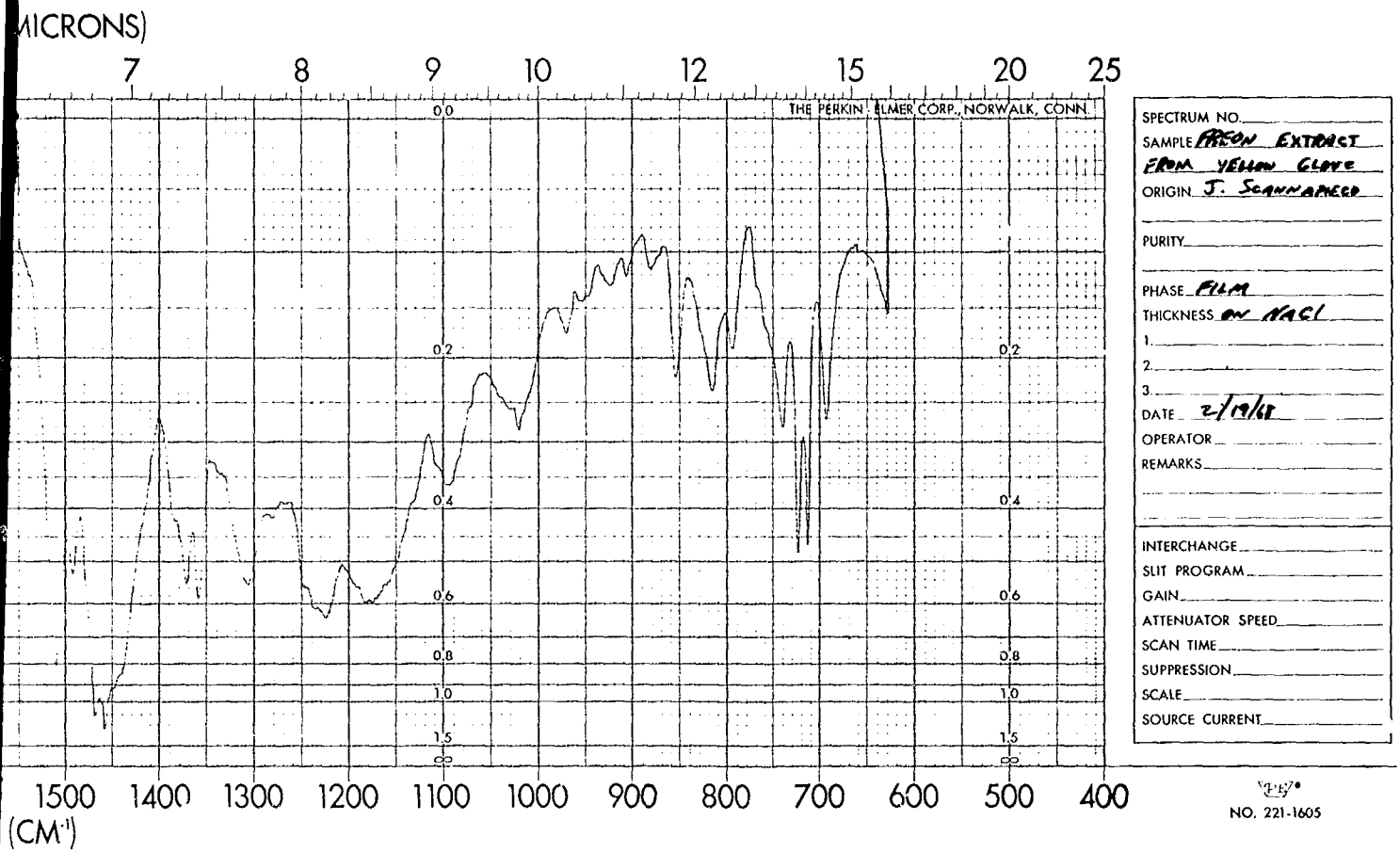
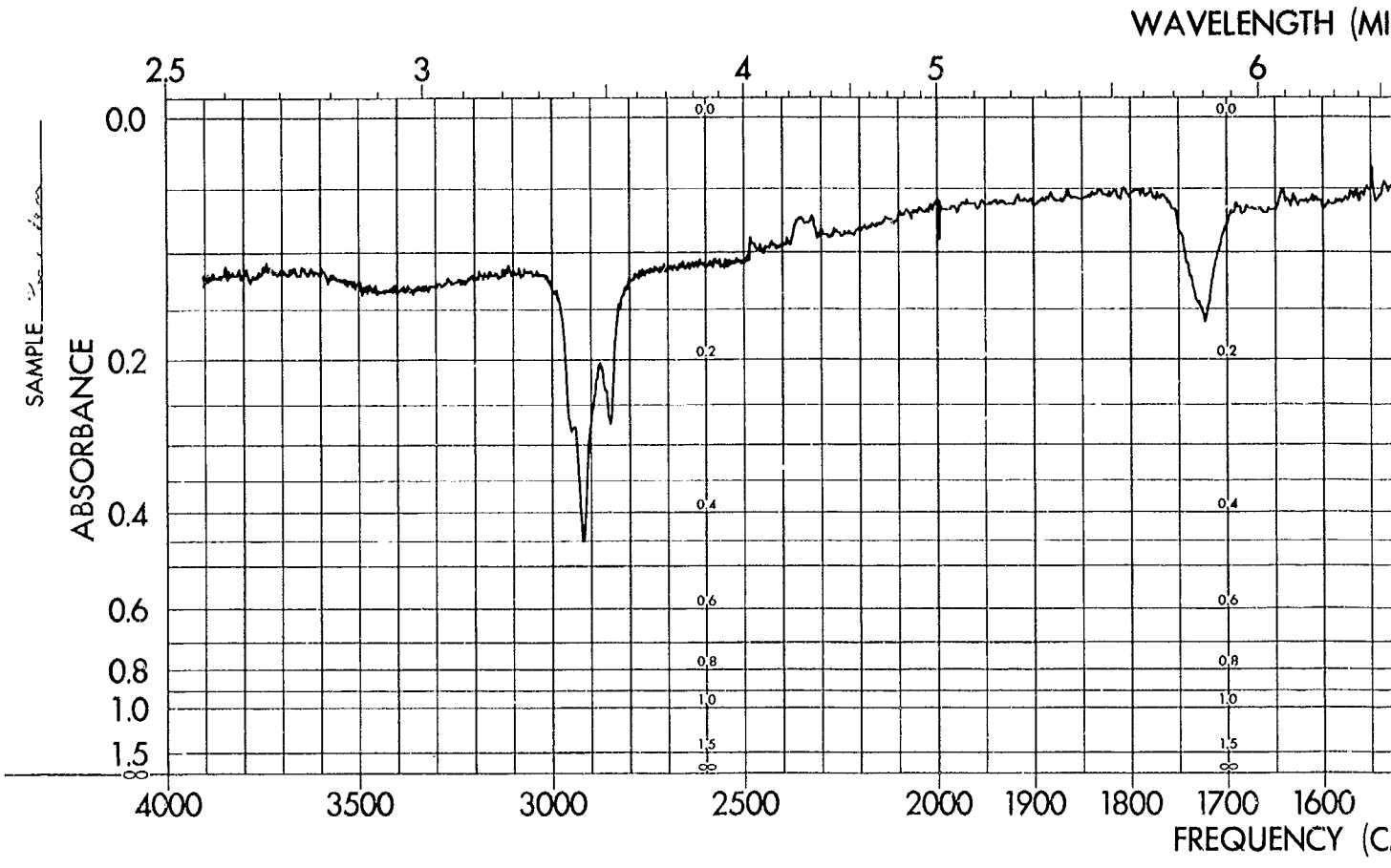


Figure 5-8. Extract From Rubber Gloves


WAVELENGTH (MI
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SAMPLE
ABSORBANCE
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FREQUENCY (C

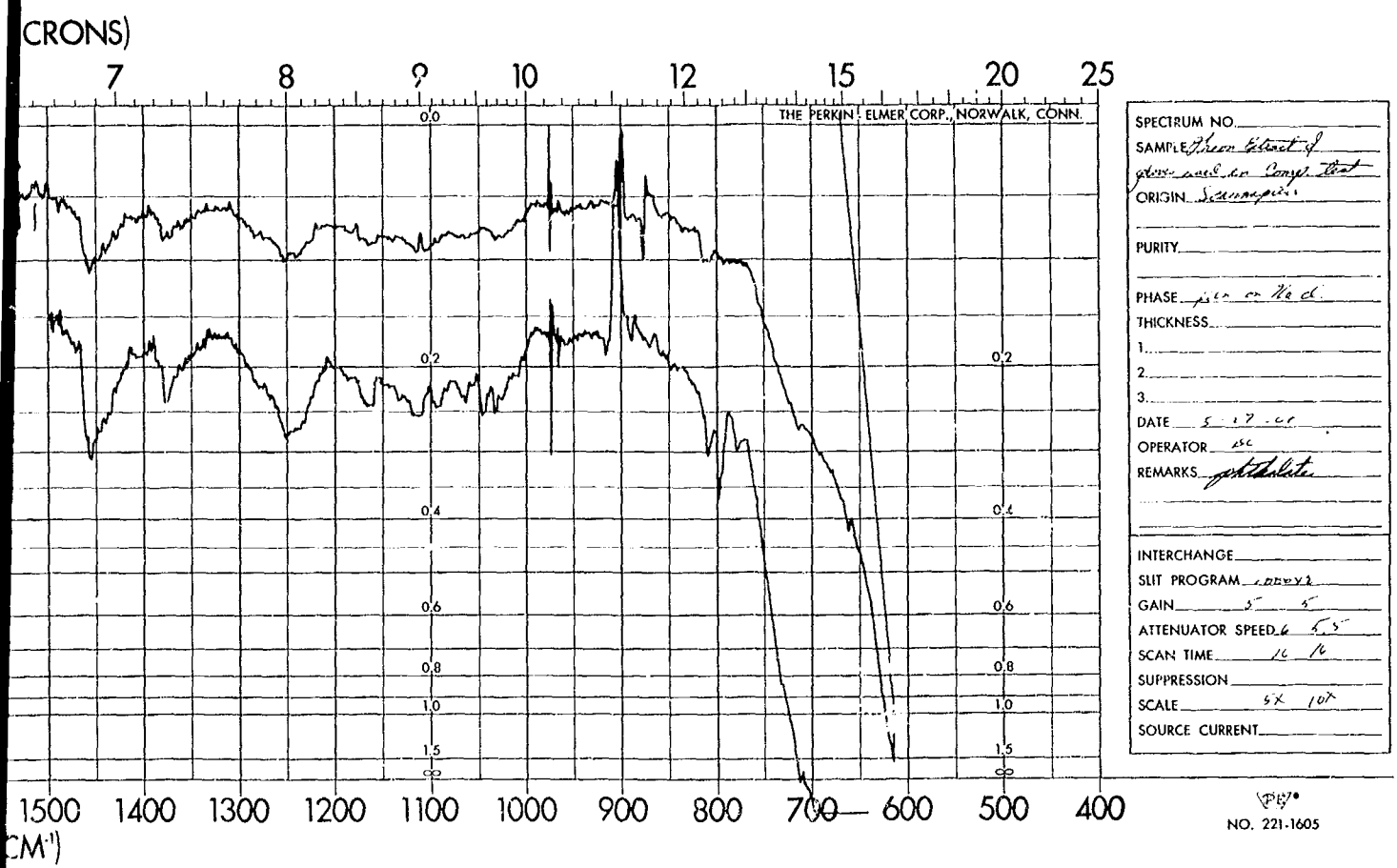


Figure 5-9. Extract From "Compar Plastic"* Gloves

*T. M. The Wilson Rubber Company

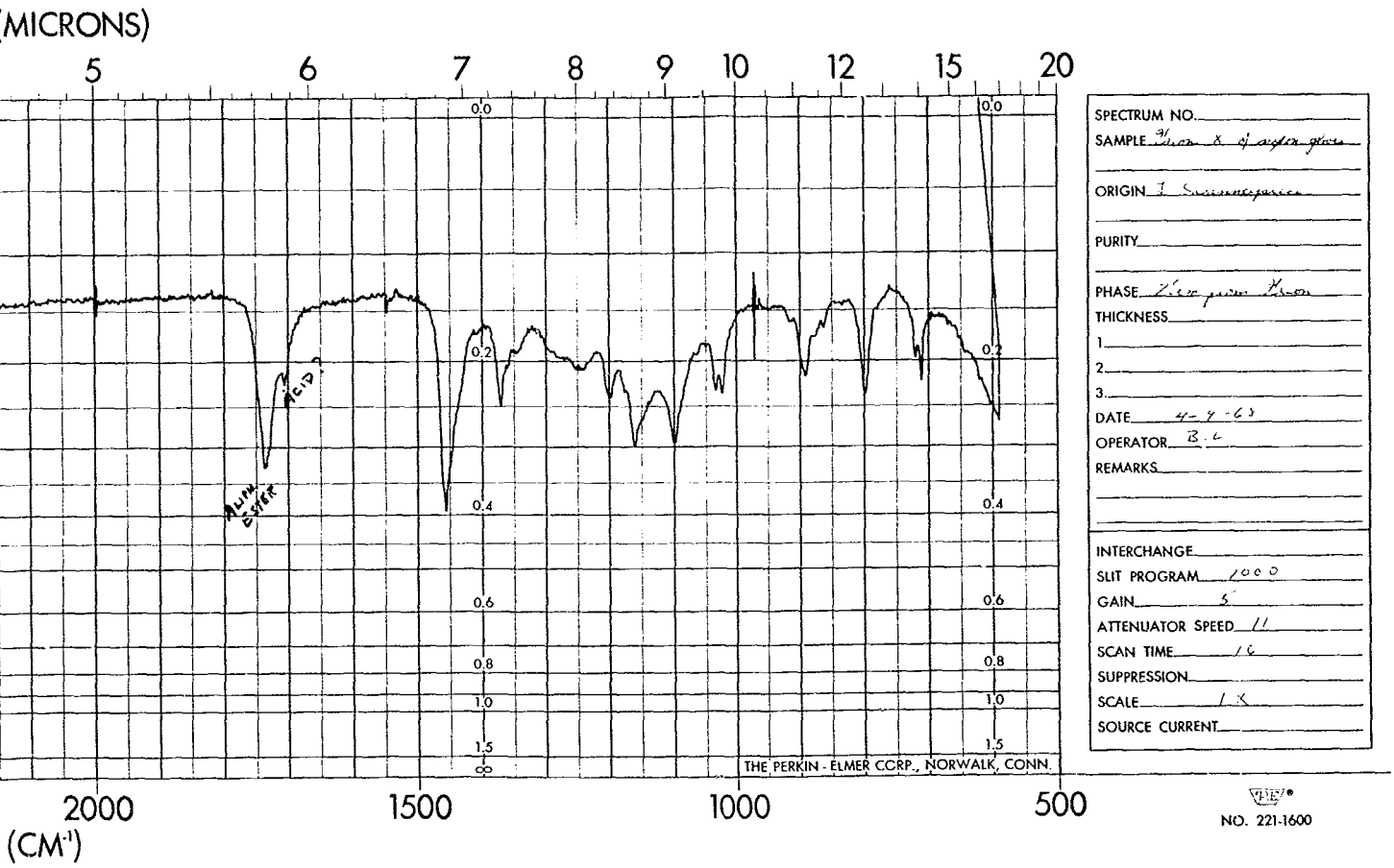
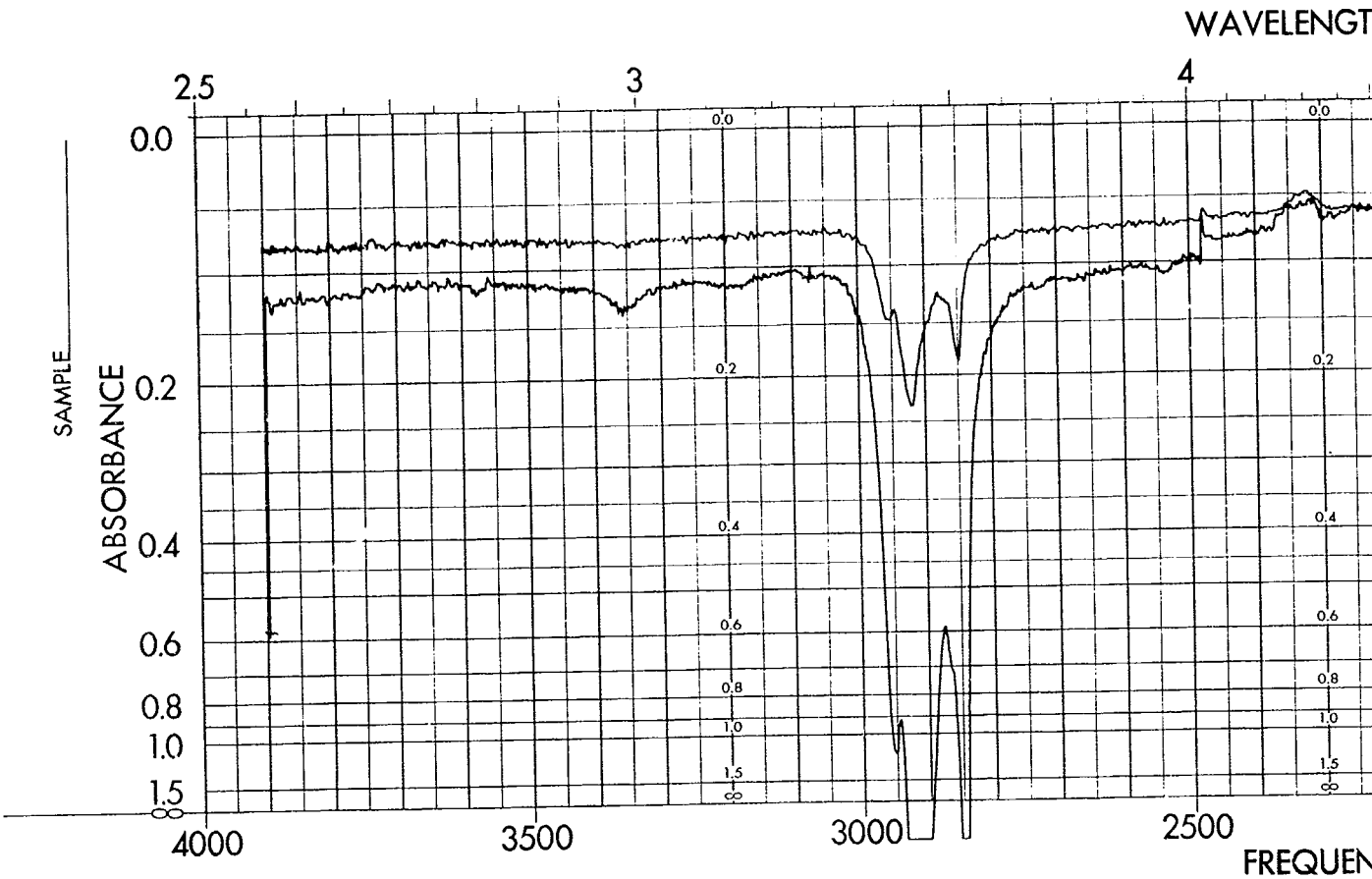


Figure 5-10. Extract From Nylon Gloves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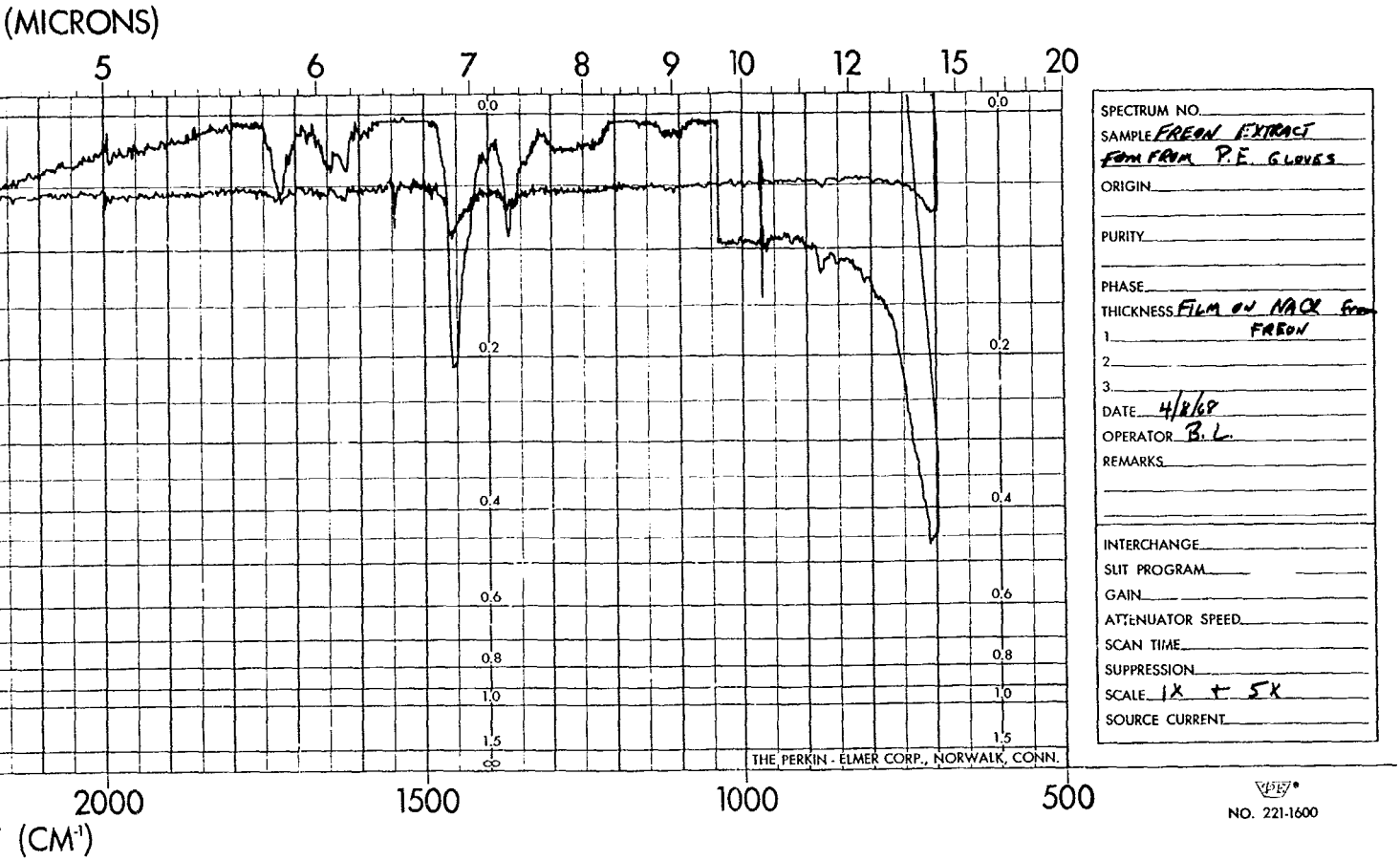
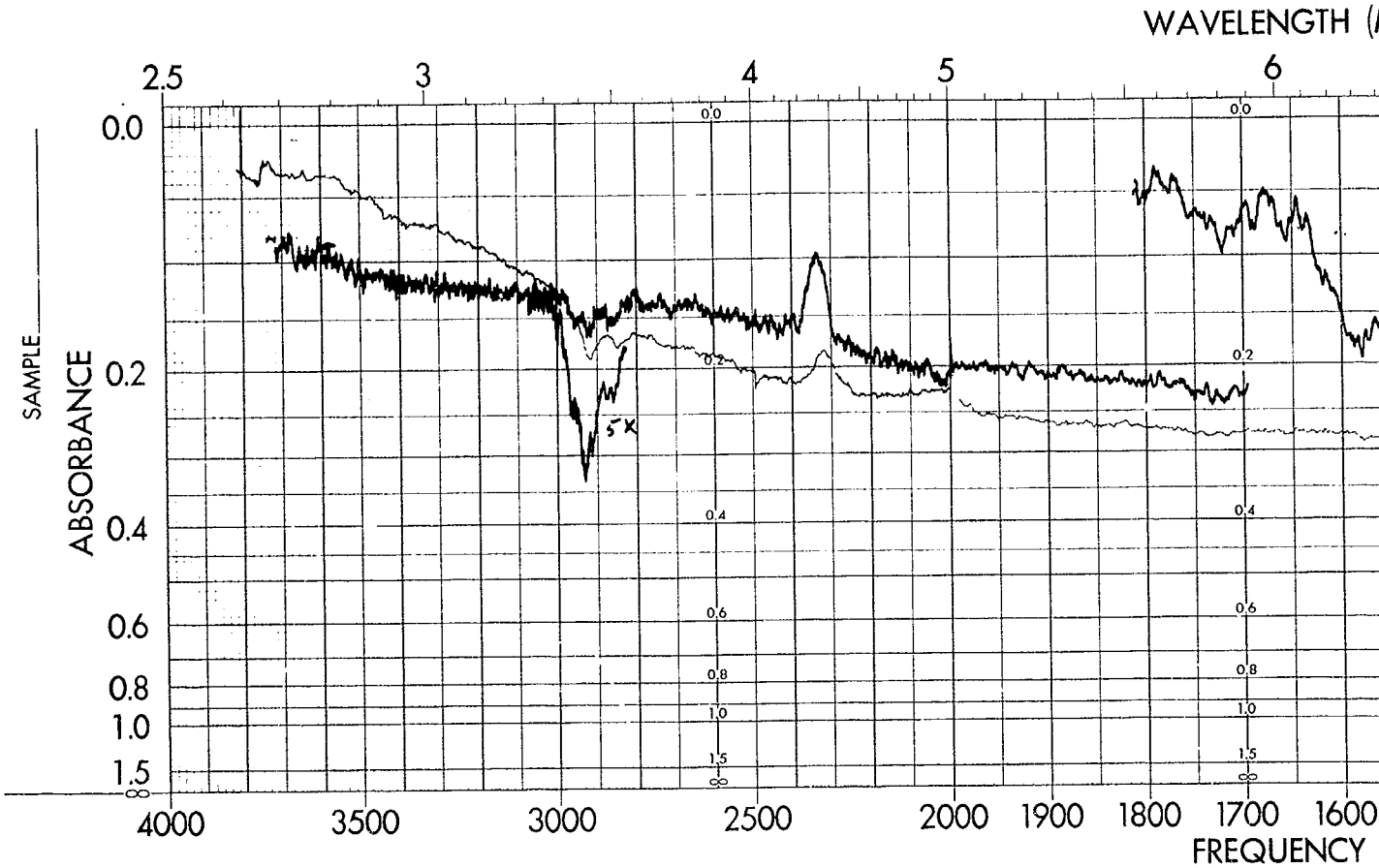


Figure 5-11. Extract From Polyethylene Throw-Away Gloves


WAVELENGTH
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6
SAMPLE
ABSORBANCE
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0.2
0.4
0.6
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1.0
1.5
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FREQUENCY

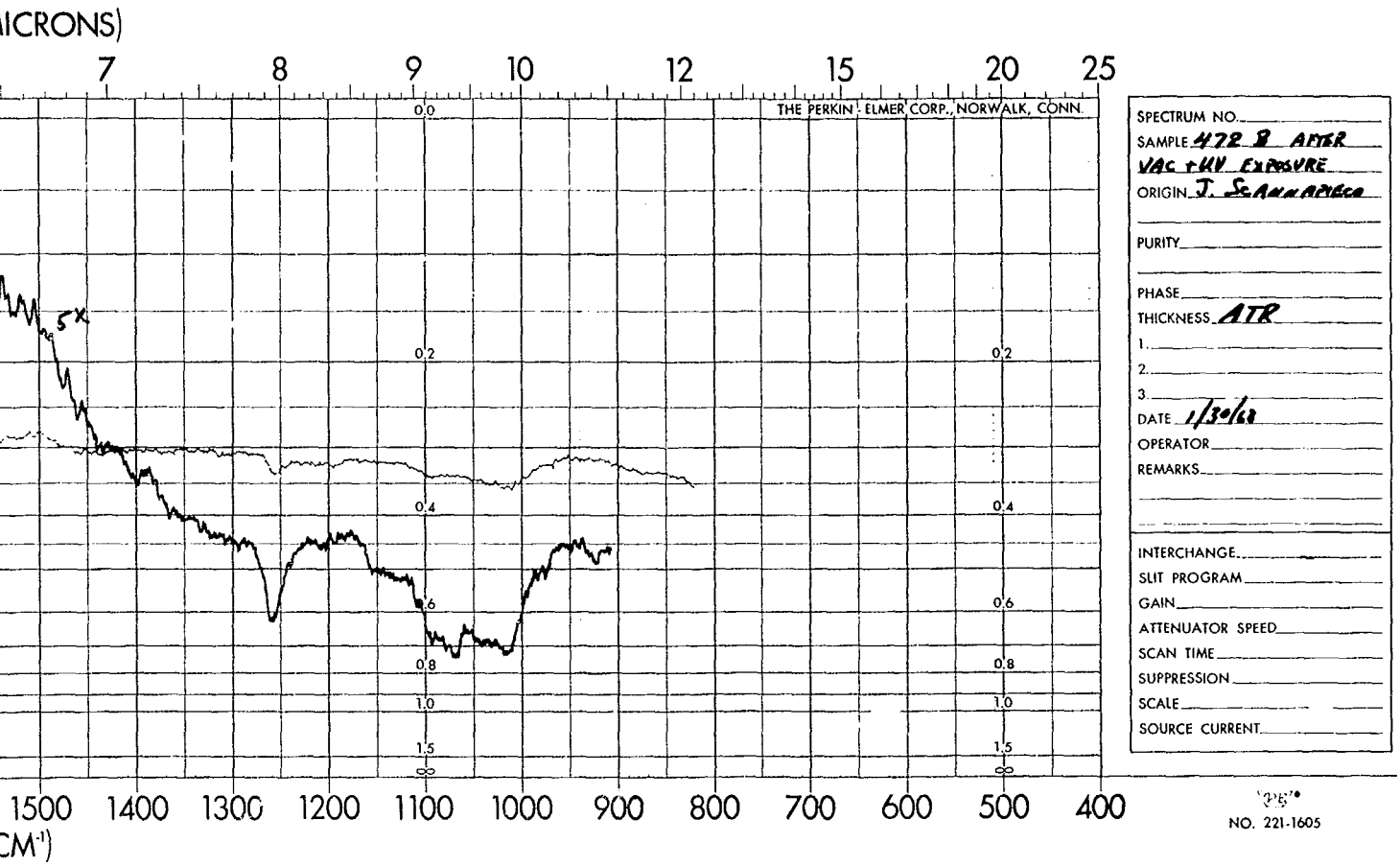
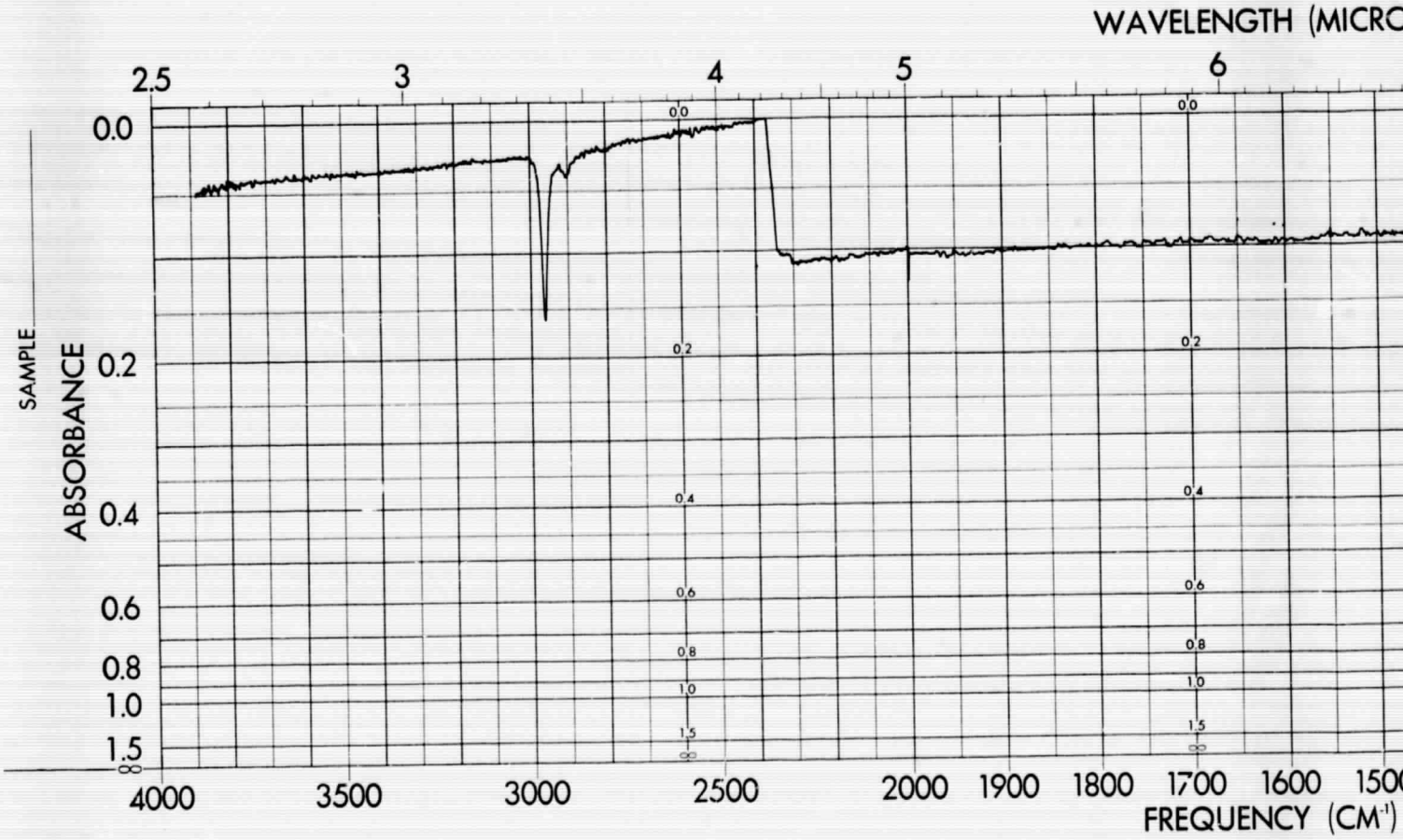


Figure 5-12. Mirror Condensate After Phthalate Elimination

FOLDOUT FRAME 1



FOLDOUT FRAME 2

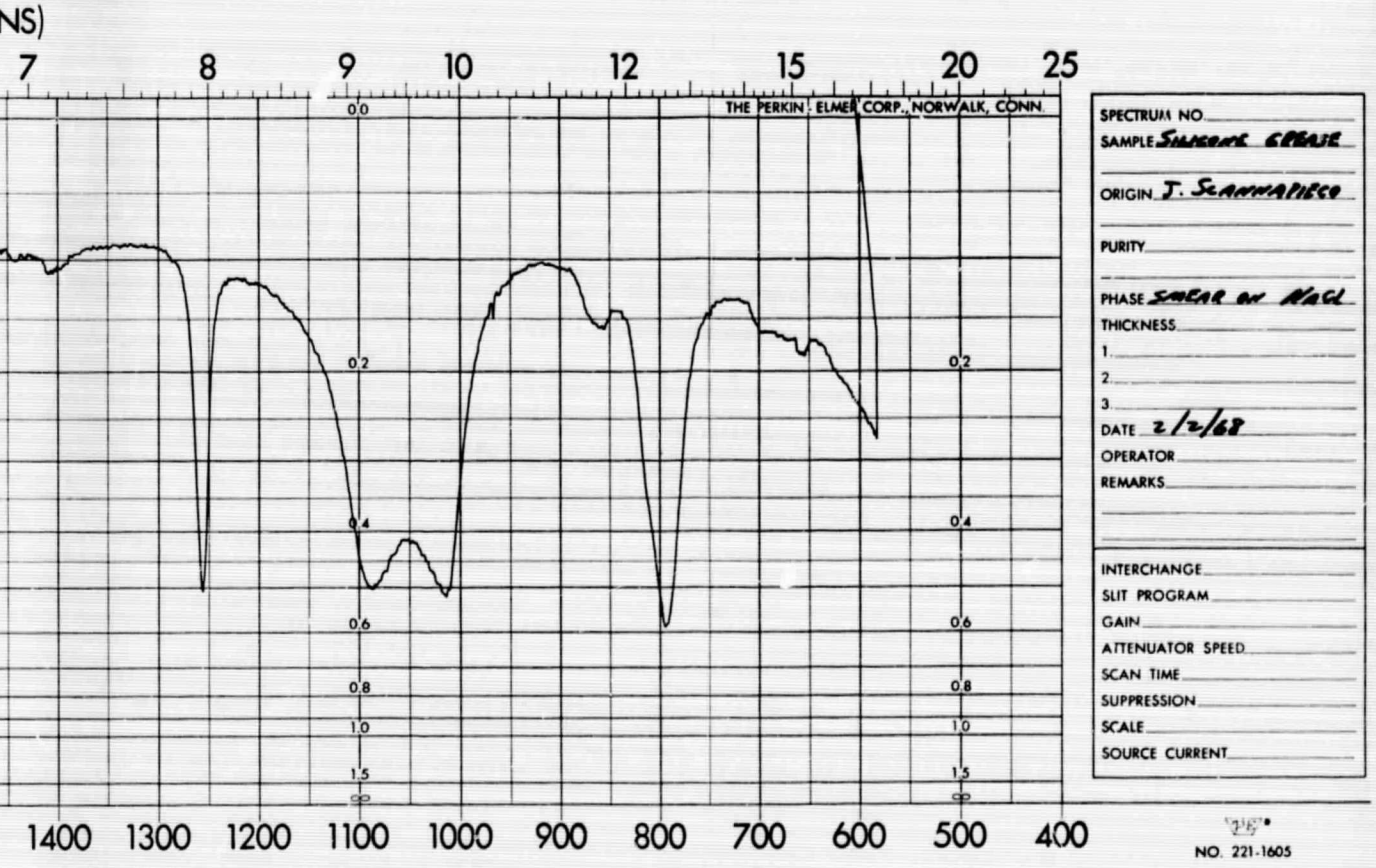


Figure 5-13. Silicone Grease

SECTION 6

CONCLUSIONS AND RECOMMENDATIONS

It has been found that there is not a direct correlation between the amount of condensate and the degradation of Lyman α reflectance (even between condensates of the same generic type of material).

Only restrictive use should be made of silicone rubbers (such as RTV-60 and Sylgard 184 tested in this program) in both spacecraft and space simulation chambers. Whenever it is used, a systems analysis considering all implications of its outgassed products on the vehicle, chamber, mission, and/or test should be considered quantitatively. This type of study has been performed with the aid of computer programs.[4]

The thin film of Mylar tested appears to be a fairly good material.

The Raychem Specification 44 Spacewire gave some visible condensate and should be investigated further before it is used extensively, because of the large amounts of wire normally used in a space vehicle or chamber.

Mirrors which are stable to this environmental exposure can be obtained. The stability of these mirrors is very dependent upon the details of the deposition technique utilized in their manufacture. This should be brought to the attention of vapor depositors and designers of optical systems. A theoretical investigation into the specific parameter(s) causing this phenomenon could aid in making vapor deposition a science rather than an art.

Based on our literature survey (Appendix B) it appears that the increase in Lyman α reflectance of the mirrors used in this study was probably due to significant aluminum oxide formation between the aluminum mirror and the MgF_2 overcoat. The techniques and equipment we have developed to perform this work have been proven suitable as they exist now. Backstreaming of pumping fluids into the chamber proper has never been evidenced by any of the analytical techniques utilized in this program. The system has shown itself to be an ultrasensitive test technique for establishing the condensability of outgassed materials. The trace amount of hydrocarbon contamination (observable only on the $-60^{\circ}C$ mirrors with

FMIR infrared scale expansion spectra and only after four days of environmental exposure) has an immeasurable effect on the Lyman α reflectance of the mirrors. A simple, proven, and economical clean-up procedure subsequent to gross condensate formation has been developed and used successfully in this program.

The outgassed products from some of the materials tested did polymerize on the mirrors. This was probably due to the ultraviolet radiation which was incident on the condenser mirrors during the four day environmental exposure.

Polydimethylsiloxane (SF-96 specifically) has a transmission window at 1216 Å. This may be dependent on the degree of crosslinking in this type of material.

An investigation should be performed to obtain gloves which both fit well and do not leach organics after immersion in cleaning solvents.

It is believed that the following information should be diseminated to all aerospace firms and agencies, this information has already been diseminated to the cognizant personnel in the Space Systems Organization of General Electric's Missile and Space Division.

a. Organic gloves should not be used with solvent on any space chamber of spacecraft cleaning or handling. All gloves investigated thusfar leach some organics (some even leave residues when used for dry handling). This is especially true when the hardware, chamber, fixturing, etc. to be handled is related to systems with critical surface properties, e.g., optics and thermal control coatings.

b. Caution should be used in application of high vacuum grease to "0" rings, drive screws, heat sinks, etc. This will be especially true if these parts are related to systems with critical surface properties. In this case it is suggested that it be eliminated.

It is our opinion that most space simulation chambers in the country today will produce condensates when subjected to the environmental exposure conditions utilized in this program if combined with the analytical techniques which we used. Even though the wall area is much larger than ours in many systems, so is the amount of source contaminats. The amount of

condensate formed by the various chambers will be a function of the amount of condensables in the chamber and the ratio of the mirror condenser area to wall condenser area in the system.

SECTION 7

RECOMMENDATIONS FOR FUTURE WORK

Now that a working facility has been put into operation and techniques for optical measurements and analysis of condensates have been proven, it is suggested that the many other materials currently used these applications be investigated.

This investigation should include the following extensions of scope:

a. Simultaneously incorporate condenser mirrors in the test which are not exposed to ultraviolet radiation.

b. Extend the electromagnetic reflectance range measured from 500 $\mathring{A}$ to about 30 microns. This range should be measured as continuous as possible, not just at discrete points.

c. Based on the data obtained in this study and other information on outgassing (some available from other programs), a quantative systems analysis of the effects of the condensables outgassed from various materials should be investigated. This can be performed on specific chambers, spacecrafts, subsystems, tests, missions, etc. using computer program techniques already demonstrated capable of performing these computations.[4]

d. Based on the recent work performed on radiation rate effects in thermal control coatings, it is suggested that real time irradiation (1 EUVS) be used in this program.

e. Consideration should be given to stopping a test after a readily visible deposit is noted on the condensers. This should include all four condensers; those under radiation, those in just thermal vacuum, those at -60°C, and those which are determining the critical temperature for condensation formation.

The order of importance, and techniques to incorporate the following extensions of scope should be investigated concurrently during the aforementioned experimental program.

a. The optical properties being investigated (reflectance, transmittance, resolution, emittance, etc.) should be measured in situ.

b. The effects of low energy corpuscular radiation, especially protons, on both the condensability of the outgassed products and on the optical properties of the condensate should be investigated.

c. Hemispheric reflectance of thermal control surfaces exposed simultaneously to the combined space environment (far and near ultraviolet radiation, low energy protons and low energy electrons) and outgassed products should be investigated. It has recently been noted that outgassed products and debris "follow" the spacecraft; therefore, this may help to account for some of the discrepancey being found between ground simulation and space flight experiments on thermal control coating degradation,

d. Other physical property measurements dependent on surface properties (e.g., resolution, scattering, etc. of optical elements) should be investigated in situ under these same environmental conditions.

e. Variable temperatures for both outgassing source and condenser surfaces should be investigated to obtain design data applicable to space chamber and spacecraft applications in general.

f. Space flight experiments should be sought for establishing correlation to both quantitative analytical and experimental studies performed on this program.

g. Angular dependance on reflectance measurements, radiation, and outgassed particle flux should be investigated.

SECTION 8
REFERENCES

1. Kovar, N.S., and Kovar, R.P., "Atmospheres Surrounding Manned Spacecraft, Sky and Telescope," March 1968.

2. Johnson, F. S., "Satellite Environmental Handbook," Stanford University Press, 1961.

3. Wright, A.N., Surface - "Photopolymerization of Vinyl and Diene Monomers," General Electric Technical Information Series Report Number 67SD290, July 1967.

4. Scannapieco, J. F., Black, J., and Tweedie, A. T., "Sublimation and Condensation of Hastelloy X in Nuclear Space Power Systems," presented at the 1968 Vacuum Metallurgy Conference, Beverly Hills, California, June 10-13, 1968 (to be published in the proceedings of this meeting by early 1969).

APPENDIX A

PROGRAM MODIFICATION TO INVESTIGATE MIRROR STABILITY TO ULTRAVIOLET RADIATION

EXHIBIT A

4-19-68

Task Description	To be Performed by
1.0 LITERATURE SURVEY	GE
Investigate the literature for observed effect of UV irradiation in vacuum of Mg F_2 overcoated aluminum mirrors and/or for developing hypotheses to explain the experimental observation of increased reflectance.	
2.0 INVESTIGATE UV INDENSITY AS A FACTOR IN PRODUCING INCREASED MIRROR REFLECTANCE	
2.1 Clean and bake out test system.	GE
2.2 Using GE "standard mirrors" (Note 1), perform a 4 to 8 day control test (Note 2) at approximately 5 EUVS (equivalent UV suns) intensity rather than 10 EUVS. Perform 1216 Å reflectance measurements before and after control test. Compare results with prior results at 10 EUVS.	GE
2.3 Concurrently with Task 2.2, check out cleanliness of test system.	GE
3.0 INVESTIGATE SUBSTRATE AND A SECOND MIRROR FABRICATOR AS FACTORS IN PRODUCING INCREASED MIRROR REFLECTANCE	
3.1 Supply GE "standard mirror" (Note 1) ferro-type substrates to GSFC for mirror preparation.	GE
3.2 Prepare GSFC mirror substrates. (Note: To be a metal substrate of GSFC selection, such as copper.)	GSFC
3.3 Using mirror substrates from Tasks 3.1 and 3.2, deposit aluminum mirror surface and Mg F_2 overcoat. Particular attention shall be given to obtaining uniformity in processing of GE and GSFC substrates.	

EXHIBIT A

4-19-68

Task Description	To be Performed by
3.4 Perform measurements of surface topography of GE and GSFC substrates produced under Tasks 3.1 and 3.2 both before and after Task 3.3. Upon completion, the mirrors produced under Task 3.3 shall be delivered to GE.	GSFC
3.5 Expose a representative sample of both types of mirrors (i.e., on GE and GSFC substrates) produced under Task 3.3 to a standard control test (10 EUVS for four days in thermal vacuum). Perform 1216 Å reflectance measurements before and after control test. Upon completion return test specimens to GSFC.	GE
3.6 Perform measurements of surface typography on test specimens from Task 3.5 using the same techniques as in Task 3.4.	GSFC
4.0 FINAL REPORT	GE
Prepare a final report incorporating the results of all work performed under this contract including the results of Tasks 1.0, 2.0 and 3.0 above.	GE

NOTE 1 - GE "standard mirrors" are defined as those used on this program to date (Mg F_2 overcoat on vapor deposited aluminum on ferrotype substrate).

NOTE 2 - "Control test" is identical to a test cycle but without a material specimen in the D-box.

APPENDIX B

LITERATURE SURVEY

APPENDIX B

LITERATURE SURVEY

A literature survey was conducted to help elucidate the mechanism of the observed increase in Lyman α reflectance. Although no direct information was found on the reflectance increase due to the type environmental exposure we used, there was appreciable information concerning the MgF_2 overcoated aluminum mirrors. References 1 and 2 discuss the effect of ultraviolet radiation on the reflectance of plain aluminum mirrors. References 2 through 7 discuss the optical properties and aging of these type coatings together with their dependence on the deposition parameters.

Based on our experimental work it appears that the Lyman α reflectance increase observed is dependent on the ultraviolet radiation component of our environmental exposure. This may even be intensity (dose rate) dependent, as evidenced by the control test conducted at 5 EUVS. References 1 and 2 discuss the reflectance degradation of uncoated aluminum mirrors due to ultraviolet radiation. They both attribute this to the optical absorption of aluminum oxide formed on the mirror. Reference 1 shows that the reaction can be reversed by irradiating in a reducing environment. If our mirrors had a coating of aluminum oxide under the MgF_2 then exposure to ultraviolet radiation could reverse the oxidation reaction and liberate the oxygen. Upon liberation, the oxygen will diffuse into the MgF_2 overcoating. Considering that the mirror is held at $60^{\circ}C$ and that it is in vacuum, it can be readily seen that the diffusion of oxygen through the MgF_2 will be much faster than the diffusion of oxygen into the MgF_2 coating under STP conditions (normal aging). Based on the known decrease in reflectance of MgF_2 protected aluminum mirrors due to aging and on the rapid reflectance increases we observed in our experiments, it is reasonable that this reaction would appear to be irreversible in our measurements. This is especially true because of the short time between irradiation and reflectance measurements. Therefore, it is reasonable that we would observe this phenomenon as a permanent increase, if it occurred. Considering the criticality of the deposition parameters required for maximum Lyman α reflectance (minimum oxidation) it is not unreasonable that we may have had an aluminum oxide film under the MgF_2. Although the reasons are not completely

understood, it is known that even the chamber size (chamber wall/substrate area ratio) is critical in obtaining the maximum Lyman α reflectance.[7] When facts such as these are considered, the existence of aluminum oxide under the MgF_2 seems more and more probable. Based on the above discussion it appears that the mirrors used in this program contained significant amounts of aluminum oxide under the MgF_2 overcoat.

REFERENCES

1. Ivanova, M.K., Lomonosova, T.N. and Yokovleva, A.V., "Action of Ultraviolet Radiation on Metallic Mirrors" Optika i Spektroskopiya, Vol. IV, Nr 4, pp. 535-536 (SSR) 1958.

2. Hass, G., Hunter W.R., and Tousey, R. "Influence of Purity, Substrate Temperature, and Aging Conditions on the Extreme Ultraviolet Reflectance of Evaporated Aluminum," Journal of the Optical Society of America, Vol. 47, No. 12, pp. 1070-1073, December 1957.

3. Hass, G., and Tousey R., "Reflecting Coatings for the Extreme Ultraviolet" Journal of the Optical Society of America, Vol. 49, No. 6, pp. 593-602, June 1959.

4. Hall, J.F., "Optical Properties of Magnesium Fluoride Films in the Ultraviolet" Journal of the Optical Society of America, Vol. 47, No. 7, July, 1957.

5. Hass, G., Hunter W.R., and Tousey R., "Reflectance of Evaporated Aluminum in the Vacuum Ultraviolet," Journal of the Optical Society of America, Vol. 46, No. 12, December 1956.

6. Fabre, D., "Variations, as Function of the Thickness, of the Reflecting Power in the Far Ultraviolet of Thin Films of Magnesium Fluoride," Acad. Sci. (Paris), Vol. 245, No. 25, pp. 2241-4, Dec. 1957.

7. Personal communication, J. Osankowski (NASA, Goddard), June 3, 1968.

APPENDIX C

TEST MATERIALS IDENTIFICATION

The following information is presented so that the materials tested are completely characterized and can be obtained again in future programs if desired.

1. NRC -2

   Manufactured by the National Research Corporation

   1/4 mil, one side aluminized, crinkled Mylar (polyethylene teraphthalate)

   Applicable General Electric Specification 147 A 1819

2. RTV - 60

   Manufactured by General Electric

   silicone rubber (polydimethylisiloxane)

   cured with 1% T-12 catalyst (dibutyl tin dileurate)

   cured for 16 hours at 150°F

   Applicable General Electric Specification 171 A 8245

3. Sylgard - 184

   Manufactured by Dow Corning

   silicone rubber

   cured with 10% part B catalyst

   cured for 16 hours at 150°F

   Applicable General Electric Specifications R6312 and 65 A 4049

4. Specification 44 Spacewire

   Manufactured by the Raychem Corporation

   Part 44/PJ6-6-23-65-4

   Wire tested 22 September 65

   roll C4A4

5. High Vacuum Grease (dimethylpolysiloxane)
   Manufactured by Dow Corning
   Applicable General Electric Specification 147 A 1803

6. Plastic Rubber Gloves (polyvinylchloride)
   Manufactured by the Pioneer Rubber Co.
   Pylox Quixams